CPE Comments and Topics

Water, water everywhere

IT is one of nature's ironies that her bounties are never scarce but only unevenly distributed. Surely the most serious obstacle facing underdeveloped countries in their drive to raise living standards is the scarcity of fresh water. In addition, consumption of water by the more industrialised countries is increasing at almost astronomical proportions. The United States, for example, is one of the countries where difficulties due to scarcity of water are already beginning to be felt. Due to her increase in population and industrialisation, a total water consumption of 240,000 million gal./day was recorded during 1960 and, as a result, many districts had to import their water from elsewhere. Such acute shortages cannot be entirely solved by strict economy and conservation because, even if new dams are to be built across rivers forming additional lakes and reservoirs, these waters will still be inadequate to meet expected demands.

Hence, desalination, or removal of salt from saline waters, becomes the only means whereby fresh water can be produced in large quantities for industry, drinking purposes and irrigation.

It must be remembered that the composition of sea-water varies extensively. In the Baltic Sea, for example, the concentration of salt in the water is 7,000 p.p.m., whereas in the Persian Gulf the concentration is 40,000 p.p.m. The average sea-water concentration can be taken as about 35,000 p.p.m. and this must be reduced to about 500 p.p.m. in order to sustain plant and human life. As far as industrial needs are concerned, the acceptable limit is nearer 200 p.p.m. because higher saline concentrations will cause serious corrosion. Several processes to remove salt from sea-water have been known for many centuries. For example, a paper in the ' Philosophical Transactions of the Royal Society ' in 1690 by Hanton described a process for making sea-water sweet for use on board ships. This consisted of distilling sea-water in an alembic and condensing the vapours with the inlet sea-water.

Present-day methods of sea-water distillation and evaporation are rather more sophisticated, but the two most important factors that have to be taken into account when estimating the economics of a desalination plant are, firstly, the amount of steam required per pound of distillate and, secondly, the number of evaporation stages necessary in order to attain a certain acceptable salt concentration.

In the U.K. both electrodialysis and flash evaporation are commercially used for water desalination. Less work is at present carried out on the freezing process. In this connection, it is interesting to recall that the Koppers Co. as well as the Fairbanks Whitney Co. have developed freezing processes. In the Koppers process, propane combines with sea-water to form salt-free alathrate crystals (1 mole propane surrounded by 17 moles of water). The crystals are separated from the mother liquor, washed and decomposed to yield fresh water. Because these crystals are formed above the freezing point of water, less energy is required than for other freezing and distillation processes. The Fairbanks Whitney method is based on a fractional crystallisation process developed by A. Zarchin in Israel (reported in CPE, 1960, **42**, 122). It would be interesting to know whether British companies will find freezing processes of sufficient economic attraction for further development.

Our special feature this month on water desalination examines this subject from various aspects. T. I. Hudson surveys current processes used for desalination such as electrodialysis, chemical treatment and distillation, discussing the advantages of each method. W. Steiner has contributed an article on the fundamental principles of design of flash evaporation plant —at present the most widely used desalination process. Finally, A. Smith describes two flash evaporation plants which are now in commercial use. This feature, we feel, will give a broad outline of current progress and thinking in this most vital subject.

More and more ethylene

THE affluence of the British petrochemical industry can be judged by the increasing production of ethylene, which is probably the most widely used petroleum chemical. There are five British companies making ethylene at present: I.C.I., Esso Petroleum Co., British Hydrocarbon Chemicals, Shell Chemical Co. and Courtaulds, producing altogether about 395,000 tons p.a. ethylene. I.C.I. are currently leading with 140,000 tons p.a. ethylene at Wilton. In addition, the I.C.I. Heavy Chemicals Division has initiated construction of a 35,000-tons-p.a. complex using ethylene to make ethylene oxide, ethylene glycol and associated products. This means that by the end of the year I.C.I. will be making almost 180,000 tons p.a. ethylene. The I.C.I. Severnside complex, now under construction, is planning to use another 40,000 tons p.a. ethylene to be supplied by pipeline from Esso at Fawley. It is felt that, if the demand is sufficiently large, Severnside will produce further quantities of ethylene by cracking naphtha (as is done at Wilton).

British Hydrocarbon Chemicals are producing 130,000 tons p.a. ethylene at their Grangemouth works. The new B.H.C. plant at Baglan Bay, which is to be completed by 1962, should substantially increase B.H.C.'s ethylene output, which will be used mainly for ethylene chloride. Shell Chemical Co. are making about 55,000 tons p.a. ethylene.

Esso have an output of 40,000 tons p.a. at Fawley using one steam cracker. All the ethylene at present made is supplied over-the-fence to Monsanto for their polyethylene and to Union Carbide Corp. for their ethylene glycol, but a small amount is also supplied to Associated Ethyl. A second steam cracker now under construction will be completed by the end of the year; this should double their output. Most of the ethylene from this second cracker will be sent to I.C.I. Severnside. Courtaulds' ethylene production at Spondon is much smaller and is mainly used for making a range of mixed alcohols. It would seem, therefore, that during the next few years the U.K. ethylene production will increase quite considerably.

Poland's chemical industry

IN common with other East European countries, Poland's plans to expand her petrochemical industry during the next decade are most ambitious. A recent article by the Polish Minister for the Chemical Industry, A. Radlinski, in *Przeglad Techniczny* pointed out that there will be a four-fold increase in the crude oil processing facilities during the next five years. One of the largest investments during that period will be the Mazovian refinery and petrochemical works which are scheduled to come into operation in 1968. Crude oil for this refinery will be supplied from the Soviet Union through the pipeline which is being built at present linking Russian oilfields with Poland, East Germany and Czechoslovakia.

According to the Minister, extensive deposits of natural gas have been discovered recently in Poland. In connection with these deposits, a licence has been purchased by the Polish Government for a process by which natural gas is converted into ammonia synthesis gas and acetylene. A large plant is to be erected at Tarnow using this process to produce an annual output of 140,000 tons of nitrogenous fertilisers, 80,000 tons PVC and large quantities of acrylonitrile and methacrylate resins. The plant will consume about 600 million cu.m. of natural gas p.a.

It is interesting to note that there are also large sulphur deposits in Poland; these were discovered in the early 1950s and are now beginning to be extracted and processed. Eventually all of Poland's sulphuric acid production should be based on local sulphur; it is also hoped in future to export some of this sulphur.

With regard to plastics production, Poland at present lags somewhat behind other European countries. It is estimated, however, that by 1965 the annual output of plastics will amount to 200,000 tons, about four times as much as at present. Apart from considerable expansion in the production of phenol and amino plastics there will be a considerable increase in the production of PVC and other vinyl polymers such as polyvinyl alcohol and polyvinylidene chloride. A high-pressure polyethylene plant should be on stream in 1964. In general, it is expected that the production of plastics per head of population should amount to 13.5 lb. by the end of this decade.

Less money for atomic energy

POSSIBLY as a result of the economic wind of change, the Atomic Energy Authority has reduced its 1961-62 estimates to £78 million compared with £93 million for the previous year. In the U.K.A.E.A. seventh annual report 1960-61, recently published, these reductions are partly attributed to reductions in uranium purchases, since the Authority now has all the uranium it will require to satisfy the current power requirements. In effect the Authority is now consolidating its gains in the gas-cooled graphite-moderated field in order to succeed in the A.G.R. and high-temperature gas-cooled DRAGON reactors. A certain co-ordination of work on reactors has been attained by the creation of a new reactor group under the capable direction of Sir William Cook.

In connection with A.G.R. fuel elements it was disappointing to read in the report that beryllium, which was considered so advantageous as a fuel canning material, is liable to considerable corrosion under high-temperature conditions.

In the past, one of the main concerns about beryllium cans was the formation of helium arising from the transmutation of beryllium when bombarded by neutrons. It was thought that helium atoms would cause brittleness and swelling of the material. However, irradiation tests in PLUTO have shown neither swelling nor increase of embrittlement. It has, however, been decided by the Authority that, until the conditions under which beryllium stands up to A.G.R. environments are fully understood, the fuel elements will not be clad with beryllium but with stainless steel. Thus a stainless steel has been developed which has better corrosion resistance to carbon dioxide than the standard 18%Cr-8%Ni alloy. Quantity production of the stainless-steel clad elements for the Windscale A.G.R. reactor has already started during the last year.

Thomson nickel project

THE first extensive nickel deposits were discovered quite fortuitously in the Sudbury district of Ontario in 1883 during the construction of the Canadian Pacific railway. At that time its uses were limited for coinage purposes, constantan (40%Ni and 60%Cu, which has an abnormally low temperature coefficient) and for electroplating. The usefulness of nickel-steel for armour plate and armour-piercing munitions was realised as far back as the Spanish-American war and from then on nickel attained a major importance as a 'strategic' metal. During the first world war the nickel industry consequently expanded considerably but found itself in a structural crisis in the 1920's.

The world's two nickel-producing companies, the British Mond Nickel Co. and the American International Nickel Co., decided to amalgamate in 1929 in order to pool their research and development activities. Since then there has been a most spectacular boom in the nickel industry. The 1935 nickel consumption was 45,000 tons compared with the 1960 consumption of 230,000 tons. Much of this increase is due to

U.S. stockpiling of nickel, which used to absorb unusually large amounts of the current international nickel output from 1947 to 1956.

One of the most modern projects for increasing nickel production was initiated last March in Manitoba, Canada. It is called the Thomson project. This is probably the most integrated minerals project in the world, comprising every stage from the mining of the ore to the production of electrolytic nickel. The annual capacity of the Manitoba mines is reputed to be in the region of 33,000 tons p.a. and will thus be the world's second largest nickel production centre. Incidentally, when the Thomson project was announced over four years ago fears were expressed that its completion would result in a threat of major nickel surpluses over a period of years. It is felt now, however, that but for the availability of nickel from the Thomson project international supplies of nickel would have run short within the next two years.

Oil in the common market

IT is extremely difficult at this stage to forecast what the effect of Britain's entry into the Common Market will have on the chemical industry. It is only possible to assess the viability and export potential of individual branches of the British chemical industry; this will show that in many cases there are both benefits and drawbacks to be gained. On the whole, it must be remembered that the Common Market chemical industry is mainly an importer of raw materials and an exporter of intermediate and finished products, similar to the British chemical industry.

As far as oil is concerned, both Britain and the Common Market countries are dependent for the bulk of their oil on the Middle East and other producing areas. Compared with current U.K. oil imports of 50 million tons p.a., West Germany imports 28 million tons p.a., France 27 million tons p.a. and Italy 23 million tons p.a. (this represents an overall increase in consumption over the last year of 17 to 25%). The Common Market countries are practically self-sufficient as regards refining capacity. Thus Italy, France and West Germany each have refining capacities in excess of 40 million tons p.a., Holland has a capacity of 20 million tons p.a. and Belgium 7 million tons p.a. For purposes of comparison the U.K. now has plant with a total capacity of about 50 million tons p.a.

In assessing the resources of the Common Market it is important to take into account the newly developed oil industry in the Sahara, which is closely linked with France. Last year's output of Saharan oil was 8½ million tons, most of which was shipped to France for refining. It is expected that this year, with the commencement of large-scale production in the Edjele area, the Saharan output should be about 16 million tons. Besides these sources a small quantity of Russian oil is already finding its way into Italy. The effect that increasing Russian oil deliveries will have on Common Market countries is still an imponderable question.

Corrosion in distillation equipment

EVIDENCE that materials present in trace quantities are largely responsible for severe corrosion in distillation equipment was put forward by W. F. Boyling at a symposium on ' Corrosion in the Chemical Industry ' held at the University of New South Wales recently. Several actual case histories involving corrosion problems associated with distillation equipment were cited by the author.

In one example a mixture of acetic acid and ethyl acetate saturated in water was passed into the column, the ethyl acetate-water coming off the top and the acetic acid being taken off from the bottom. A by-product stream containing ethyl acetate, acetic acid, traces of ethyl formate, formic acid and other organic materials was available from another part of the site and was added to the ethyl acetate circuit. It was noticed that each time the column was shut down for cleaning a deep blue-coloured liquid was drained from the reboiler. Inspection of the lower section of the column showed that some bubble caps had been dissolved whilst others were badly corroded.

The main sources of corrosion were thus attributed to traces of formic acid and the problem was solved by replacing the mild-steel column by 18/8/3 molybdenum steel.

In another plant producing acetic acid/acetic anhydride mixtures by acetaldehyde oxidation in a reactor, the reactor products containing acetic acid, acetic anhydride, water, unreacted acetaldehyde and catalyst were fed to an 18/8/3 molybdenum steel distillation column just below the mid-point. Water, acetic acid and acetaldehyde were taken off as overhead product whilst the acetic anhydride and catalyst were taken off the base. Severe corrosion was found to occur at a short distance below the feed point.

Two approaches were made to overcome this problem. Firstly, by making this section of copper its lifetime was found to increase by more than six months. Test strips of *Hastelloy* C in the column showed less than half the corrosion rate for copper and tantalum test strips proved even more successful. The second approach, carried out simultaneously with the first, was to determine the cause of this corrosion. Evidence showed that water was present in small quantities in the corroded section itself—below it the anhydride was dry and above it the water concentration was quite appreciable. In addition it was found that some catalyst (metals from Groups VII and VIII) was required for corrosion to occur.

To overcome corrosion, the reactor was redesigned so that all the catalyst remained behind in the reactor and the products were taken off as a vapour through mist separators. A flash evaporation circuit was installed between the reactor and the separating column which was then fed with mist-free vapour. Finally an organic ester entrainer was added to the system and the drying out occurred under lower temperature conditions and at a different concentration of the corrosive components.

Bacteria for mineral extraction

ONE of the more interesting projects carried out by the National Chemical Laboratory was recently referred to in the 1960 N.C.L. annual report. On behalf of the U.K. Atomic Energy Authority the N.C.L. examined the influence of experimental variables on the rate of leaching uranium from columns packed with mixtures of uranium ore and pyrites. Results have suggested that the rate of uranium leaching is controlled by the rate of pyrite oxidation rather than the diffusion rate of solutions within ore particles. It was found that the presence of bacteria has a marked influence on leaching rates; for instance, only 2% uranium was extracted from a sterilised ore-pyrite mixture after 10 weeks' watering, whereas uranium extraction from an unsterilised control column after a similar time was 35%. The influence of oxidising bacteria on leaching rates is further shown by the fact that at 45° to 50°C. there was a sharp decrease in leaching rate and lowering the temperature to 35°C. markedly increased leaching rate. This may be explained due to the activity of the particular oxidising bacteria, *ferrobacillus ferro-oxidans* and *thiobacillus thio-oxidans*, which are inhibited above 40°C.

Similar work has also been reported by the Kennecott Copper Corp. in U.S. Patent 2,829,964. In their process the oxidising activity of bacteria was harnessed to recover copper from mine waste dumps. The naturally occurring species *ferrobacillus ferro-oxidans* was specially bred to withstand 17,000 p.p.m. zinc and 12,000 p.p.m. copper as well as smaller quantities of other metallic substances. In a leach solution containing a low proportion of copper and a high proportion of iron these bacteria oxidise ferrous iron to ferric, which in turn oxidises the cuprous to the cupric state and builds up a concentration of this copper in the leach solution, from which it can be readily recovered. The bacteria are autotrophic and require oxygen and carbon dioxide for growth but no organic materials. They exist in, and are tolerant to, sulphuric acid. This is surely a remarkable application of bacteriology to chemical engineering; in fact, biochemical engineering.

Industrial radiation

NOWADAYS, when we must take a saner, more money-conscious view of industrial radiation processes, it is all too clear that the possibility of wide applications for radiation processing is connected with the belief that the cost/kWh of radiation energy can be brought down to an attractive level. However, despite over a decade or more of research and development work, ionising radiations have not become a significant industrial source of process energy.

Nevertheless, according to S. J. Stratis writing in the April 1961 issue of *Research Reactor Journal*, in the U.S. about a dozen processes involving radiation energy have been reported to be out of the industrial laboratory stage; these are at present in various phases of process development and test production. Most of these processes have applications in the plastics industry; others have applications in petrochemical synthesis and product sterilisation. In chemical and petroleum companies it appears that radiation synthesis of chemicals faces stiff competition from conventional processing agents. It is not expected that the radiation refining of petroleum will occur until the cost of radiation is brought down at least to a few cents/kWh. It is quite possible that alkylation processes may be the first application of lower-cost radiation in the petroleum industry.

According to present-day knowledge, no chemical product can be synthesised with radiation. Whether radiation will ever produce a truly unique product will depend on the results of future research, such as on the properties of newly identified radiation-induced species. The ability of radiation to produce free radicals at low temperatures is already applied by industry to chlorinate benzene for the production of the insecticide lindane. In this case, ultra-violet light has sufficient energy to dissociate the chlorine molecules into the corresponding free radicals which saturate the benzene ring by a chain mechanism.

How much oil?

THE perpetual question that is asked by our more far-sighted planners is 'when will the world's oil supplies run out?'. Even at the end of the last century one eminent geologist was predicting that within a few years known oil reserves would be exhausted and it must be remembered that consumption of oil at that time amounted to only 1% of present-day needs!

One simple method of predicting how long these reserves will last is to divide them by the annual consumption. Using similar calculations it is also possible to make allowance for any probable increase in world demand in the future and so to ascertain a probable date when present-day reserves will have been exhausted. It is not difficult to imagine how serious would be the effect on the world economy if an exhaustion of world supplies did occur and for this reason those industries which are at present responsible for providing oil have also devoted considerable effort to finding new resources. During the last 20 years, for instance, the Middle East oilfields have been greatly expanded, the principal Canadian oilfields have been discovered and unbelievable reserves have been located in North Africa.

Together with this there has been a constant improvement in production technique. One new innovation is the development of 'secondary recovery' methods by which gas and water are injected at an early stage into the oil-producing formation, in order to maintain pressure in the field and assist the maximum flow of oil over a long period. Another very interesting technique is known as 'fire drive' or '*in situ* combustion'. This involves heating the crude oil below the ground in the formation where it is contained.

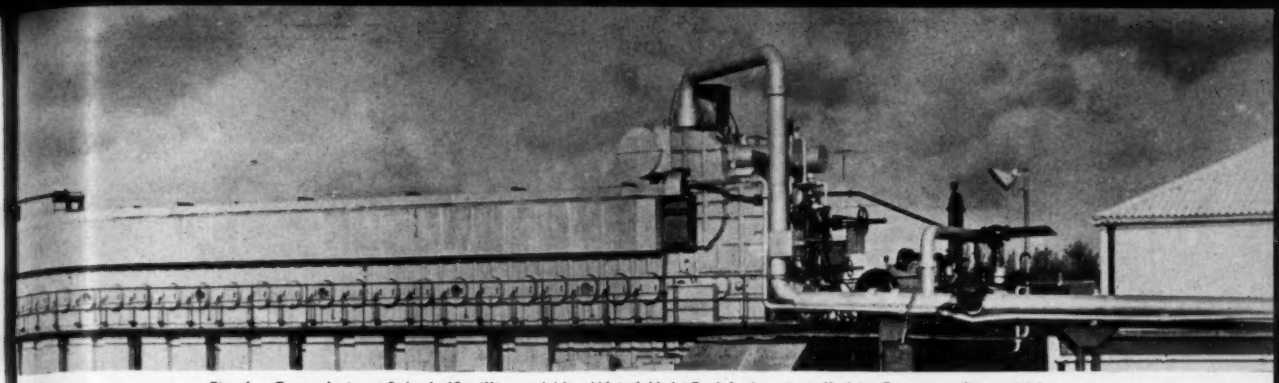

Fig. 1. General view of the half-million-gal./day Weir 'Multiflash' plant installed in Guernsey, Channel Isles

Fresh Water from Saline Sources

By T. Irvine Hudson, M.I.Mech.E., M.Inst.W.E.

The uneven distribution of rainfall throughout the world has created many regions which suffer from an extreme shortage of water, both for drinking and industrial purposes. During the last decade many methods have been economically developed for reclaiming fresh water from brackish or saline sources. This article surveys such existing methods, which comprise mainly chemical reaction processes, electrodialysis and distillation. The suitability of each method for a particular application is discussed.

THE three essentials to human existence are water, soil and energy, in that order of precedence and, from the earliest days of man when needs were limited to human consumption and the irrigation of crops, the availability of water supply with the minimum expenditure of energy was always a prime factor in selecting the locations for human habitation.

Many of these early settlements eventually developed into the industrial districts of today, since there is hardly any industrial process which can survive without water. It is not generally realised, for example, that approximately 400 gal. of water are needed in the various processes of producing one motor-car. Today, when new areas of manufacturing industry are envisaged, the availability of water is of prime importance in selecting a suitable location.

All water originates from the sea, from which the clouds are formed by solar evaporation and, in turn, produce the world's rainfall, but the distribution of rainfall on the land areas is not always proportional to man's needs, and this natural evolution needs to be supplemented by artificial means in areas of acute deficiency.

In industries concerned with the extraction of natural wealth from the earth, the location must be at the site where such wealth exists, which is not necessarily a good place for water. Examples of these are the gold and diamond mines of Africa, the tin mines of Malaya and, more particularly in recent years, the greatly accelerated development of oilfields in many arid countries.

Potable water supplies in such cases, while no less important, are frequently deficient or non-existent in the natural state, but there is often an ample supply of brackish water from mine workings, and there are also the limitless quantities of salt water from the oceans of the world. The problem is to convert these natural liquids into a quality suitable for domestic and industrial use.

Again, many islands of the world have either deficiency of rainfall, or the topography of the land area is unsuitable for the construction of impounding reservoirs, except at prohibitive cost. The strata may be of a nature which precludes natural underground conservation, and other means need to be found to supplement the natural fresh-water supply. Small quantities of drinking water for emergency use in aircraft dinghies and ship's lifeboats are essential for the preservation of life at sea.

There are three well-established methods by which these various needs can be met:

(1) The chemical reaction process for relatively small quantities required for emergency use.

(2) The electrodialysis process for emergency use at sea, and also for the production of fresh water in bulk from brackish sources.

(3) By distillation for the conversion of sea-water in bulk.

Chemical process for emergency

Survival at sea for shipwrecked sailors, for which the first essential is water to drink, has been a problem since ships first sailed the seas. A limited supply in casks was always carried in lifeboats. In recent years, the hazard has extended to aircraft passengers and crews in like circumstances, the casks having been replaced with water in sealed cans.

Development of a simple chemical process for desalting sea-water ensures drinking water supplies for long after the initial canned supplies have become exhausted; in fact, these can be

dispensed with altogether and the process relied upon entirely.

The design of suitable equipment was greatly accelerated during the 1939-45 war, notably by the Permutit Co., who produced a compact apparatus. The packs, which are easy to store and use, consist of a storage bag and a purifier bag, both of flexible rubberised material, together with a number of charges of silver-barium zeolite compressed into cubes. The kits are obtainable in two sizes for producing 2 or 6 pints of drinking water and weigh respectively ¾ lb., and 2 lb. So long as sufficient cubes are carried, sea-water can be converted into drinking water daily over long periods.

Each chemical charge comprises four cubes double wrapped in heat-sealed cellophane, and will produce ⅔ pint of drinking water from sea-water having a salinity of 35.5 g. per 1,000 g. (standard sea-water).

To operate the equipment, a metal plug is inserted in the purifier bag outlet tube. The bag is then filled with sea-water to an indicated level, and the appropriate chemical charge added. After 5 min. the contents are kneaded, and then shaken occasionally during a period of 30 min. The reaction between the chemical charge and the dissolved salts in the water is then complete, and clear drinking water can be squeezed through the outlet tube into the mouth or into a container. Residual solids and salts are retained by a filter pad in the purifier bag, these deposits being rinsed from the bag before the next desalting operation.

Within recent years, this apparatus has been carried as the sole equipment for drinking-water supplies on some ocean voyages by small sailing vessels.

Electrodialysis

The idea was thought of in the late 19th century, but the lack of suitable materials to use with it prevented its economic development at that time.

Electrodialysis is a means of removing salt from water, as opposed to distillation which removes water from a salt solution. Fundamentally, the process depends upon the fact that saline water is capable of conducting a current of electricity, due to the fact that, when sodium chloride is dissolved in water, it immediately dissociates into sodium and chlorine ions. Sodium ions are positively charged, while chlorine ions are negatively charged.

If, therefore, two electrodes having a potential difference are placed at opposite ends of a tank of salt water, and a direct current of electricity is passed between them, the sodium ions will move towards the cathode and the chlorine ions will move towards the anode.

If two diaphragms having no ion exchange properties, but permitting ion travel without bulk movement of the water, are placed between the electrodes and a current passed, the chlorine ions will concentrate in the anode compartment, the sodium ions in the cathode compartment, and the centre compartment will become desalted.

Further development of the principle has become possible by the production of plastic-based ion-exchange membranes, which have the effect of considerably reducing the amount of electrical energy required, and making the process economically sound on a commercial scale.

These membranes are available in various forms, but fall into two broad categories: the heterogeneous type with a matrix of some inert material such as polyethylene in which is embedded active material such as ion-exchange resin; or the homogeneous type made by activating an otherwise inert material to give it ion-exchange properties. They are made as positive membranes permeable to negative ions, and negative membranes permeable to positive ions.

Thus, by multiplying the number of cells in a given plant, within certain practical limits, with the electrical potentials of the membranes alternating throughout the stack, alternate diluate and concentrate compartments will thus be formed and plants of varying output can be made. Pumps are incorporated with the plant to maintain circulation through the respective diluate and concentrate compartments; a conductivity cell indicates when the water has reached the required stage of dilution, the concentrate being periodically blown down to maintain predetermined limits of concentration. Fig. 2 is an example of an electrodialysis desalting plant produced by the Permutit Co. Ltd.

The probable limit of raw water salinity which can be dealt with economically by electrodialysis is in the order of 15,000 p.p.m., which covers a considerable range of the land-based brackish waters of the world, and for such cases is generally as cheap, or cheaper, than distillation.

William Boby & Co. Ltd. installed in Tobruk some 2½ years ago an experimental plant treating a water containing 4,800 p.p.m. of salt, and originally designed to produce some 2,500 gal./day of fresh water. This was the first plant built by any European country which was larger than laboratory size. Following installation on the site, further development work was carried out during the first three or four months, and it was eventually found capable of producing over 12,000 gal./day, at which output it has been working satisfactorily for the past two years.

As an example of the rapid progress being made in this field, the South African Government, in conjunction with the Anglo-American Corp., have nearly completed a large electrodialysis de-salting plant for the Geduld Gold Mines in the Orange Free State, which has a capacity of 2.4 million gal./day. In this case they are using a new type of parchmentised membrane developed in South Africa.

Emergency use

For emergency use, such as producing the modest quantities of fresh water from sea-water required for survival at sea, the economics of the process are of lesser importance and are largely discounted.

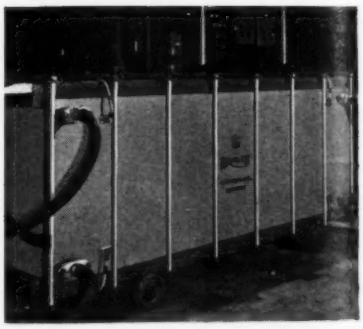

Fig. 2 'Permutit' electrodialysis unit

A simple apparatus using this principle for aircraft dinghies and ships' lifeboats has been developed by N.V. Bronswerk of Amsterdam. The plant, which is known as the *Salvator* 10, is of compact dimensions to permit easy location underneath one of the cross-benches at the bottom of the boat. It is designed to produce 2 gal. of drinking water every 3 hr., or 24 gal./day, from ordinary sea-water.

It mainly comprises one membrane stack, having an equal number of cationic and anionic membranes, assembled to provide alternate diluate and concentrate cells, which form parts of separate circulatory systems. The stack is mounted in dry condition into a housing which ensures a sealed construction frost and tropical proof. Two manually operated semi-

rotary pumps made of sea-water-resistant material are provided for the circulation through these systems. Power is obtained from a hand-operated direct-current electric generator, provided with handles for operation by two men at the same time. Interconnecting piping and fittings, together with a 3-gal. storage tank, are all made in PVC.

Although in practice the quality of the drinking water will be mainly determined by taste, it is possible to check it more accurately by means of an ammeter fitted to the unit. The complete assembly weighs approximately 98 lb. Assuming that there are 50 people in a lifeboat, this plant can, if necessary, provide nearly $\frac{1}{2}$ gal./day/person of fresh water—an ample quantity for survival at sea.

Distillation

The production of drinking water from sea-water by distillation has been practised for many years. The first marine evaporators were installed in ships about the year 1870, and during the 1914-18 war many were installed as land-based units to supply drinking water to troops operating in the waterless deserts of the Middle East.

These evaporators were generally of the submerged-tube type, where the brine is pumped through a circular cast-iron vessel, at about half-full level, and boiled with a submerged steam coil. The brine vapour is then led to a condenser, the tubes of which are cooled by the incoming brine, and de-aerated with a high-pressure steam air ejector.

To effect further economy in steam consumption the number of brine vessels are multiplied. The brine vapour from the first vessel containing the steam coil is led through a coil submerged in the brine of the second vessel and so on through successive stages to the final condenser. Incoming sea-water is in turn gradually heated by passing it through coils in the vapour spaces of each brine vessel, finally entering the first vessel containing the live steam coil.

There are, however, a number of limitations to this type of plant, the first of which is capital cost and high shipping cost, owing to the bulk of the brine vessels. They are of necessity made in cast iron for corrosion resistance and, while scale formation is largely overcome by the use of ferric chloride, its cost is still unduly high. Owing to the small end temperature difference on the heat exchangers as the number of brine

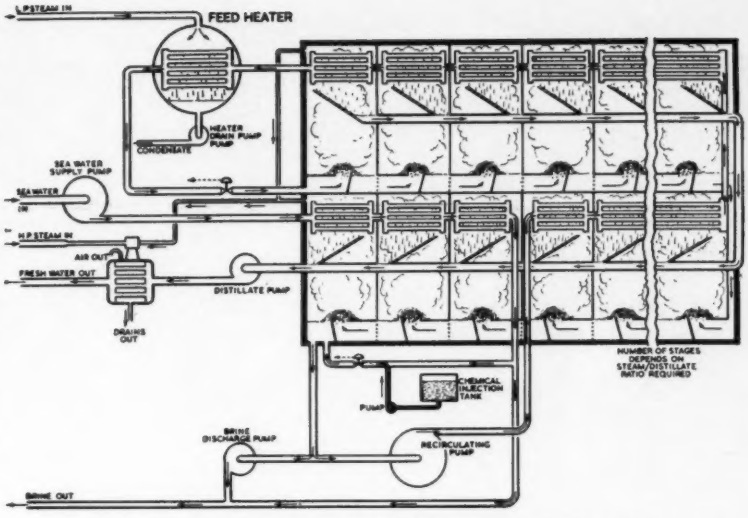

Fig. 3. Flow diagram of Weir 'Multiflash' sea-water distillation plant

vessels is increased, the limit of efficiency was generally in the order of 8 to 1, water/steam ratio; most plants of this type were designed to operate on a 6 to 1 water/steam ratio.

Further progress, therefore, depended on an entirely new conception of the process, as a result of which the 'flash' evaporator has been developed. Briefly the principle is to heat the brine in a series of tubes, at a pressure above the local boiling point, and then pass the heated liquid through a series of flash chambers under vacuum conditions, in which the flashed vapour is condensed, de-aerated and collected as a distillate of high quality for domestic and industrial use.

Fig. 3 shows a simplified flow diagram of a multi-flash evaporator manufactured by G. J. Weir & Co. Ltd. In this type of plant, an efficiency of up to 12 to 1 water/steam ratio can be attained, corrosion and scale formation are so reduced by adequate aeration that only a minimum use of chemicals is needed to ensure continuous operation over long periods, and units of compact size can be fabricated in mild steel. The plant can thus be made at much reduced capital and shipping cost, with greatly improved operating efficiency.

The risk of scaling and corrosion has been substantially overcome by the flow design, the sequence of which will be clear on reference to Fig. 3. The sea-water feed combined with surplus cooling sea-water flows through the primary heat exchangers. Surplus cooling water is then rejected and chemical treatment applied at this point. The feed then passes to the first flash chamber, where it joins the main flow. Of the latter, the larger part is recirculated, and a quantity blown down to keep the concentration within predetermined limits.

On entering the first flash chamber, the feed comes into intimate contact with the flashed vapour rising from the brine inflow from No. 2. De-aeration of the raw water feed thus takes place, the released gases (mainly oxygen and dissolved carbon dioxide) being drawn off by the steam-jet air ejector, which creates a high vacuum in No. 1 flash chamber and creates progressively lower degrees of vacuum in the other chambers.

The recirculation quantity is then pumped through the secondary heaters to the external steam heater, throughout which operation the pressure is maintained above the local boiling point. Flow continues through a control valve, pre-set in accordance with the desired distillate output, to enter the final flash chamber, where the reduction of pressure causes formation of vapour and release of dissolved gases. These are removed through an orifice pipe by the main air ejector.

The distillate cascades down through all the flash chambers, the total being withdrawn by the distillate pump, passing on the way to storage through the air ejector, thus condensing the high-pressure steam used to create the necessary vacuum.

Fig. 1 shows an installation by G. J. Weir & Co. Ltd. for the States of Guernsey Water Board, having an output of 500,000 gal./day, with a performance ratio of 11.6 to 1 fresh water/

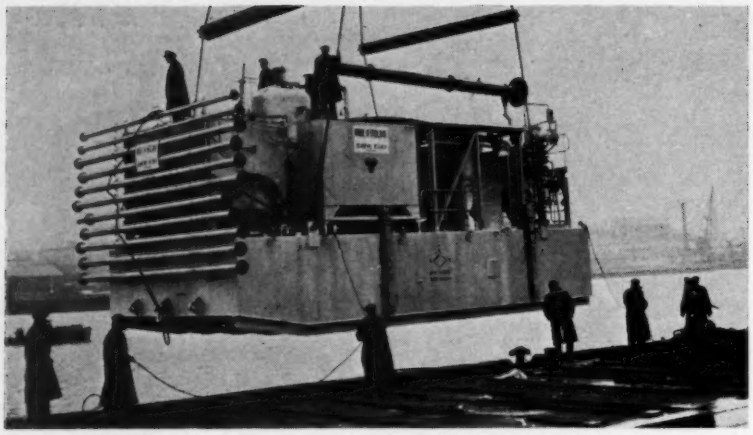

Fig. 4. A 14,400-gal./day sea-water evaporator built by Richardsons, Westgarth & Co. Ltd., being floated down river, destined for Abu Dhabi in the Persian Gulf

steam. This was installed to meet the increasing demand on the island's fresh-water supply. Additional impounding reservoirs for the further conservation of rainfall would not only have entailed very high capital cost due to the topography of the island, but would have occupied valuable agricultural land. This was, therefore, a case in which distillation of sea-water was the more economical method of supplementation.

Another plant has been constructed by the same company for the Government of Kuwait, with an output of 2 million gal./day of fresh water from sea-water. Evaporators of the submerged-tube type have been producing 1.12 million gal./day during the past five years. The new plant will therefore increase this to a total of over 3 million gal./day of fresh water from sea-water, in a desert area where natural fresh water is virtually non-existent.

A new plant of novel design, shown in Fig. 4, has been recently completed by Richardsons Westgarth & Co. Ltd. for the sheikdom of Abu Dhabi on the Trucial coast, having an output of 14,400 gal./day of fresh water from sea-water. Hitherto, no supply of fresh water has existed at Abu Dhabi, all having to be imported by dhow at considerable expense.

A particular problem facing the designers was the complete absence of any port facilities at the destination, local fishing vessels providing the only means of transporting cargo from ships anchored off shore. The evaporator, of the vacuum flash type, together with its boiler, diesel alternator set, pumps, fuel tanks and other accessories, was therefore mounted as a self-contained unit on its own pontoon. The illustration shows it being lowered into the River Tyne, to be towed down river to the cargo ship, the sea-water suction and discharge pipes being mounted as shown for convenience in transit.

The complete unit, taken as deck cargo, was lowered into the sea some miles off the Trucial coast, towed to the beach with the aid of a winch, an integral part of the plant, and hauled on to a prepared foundation. It was only necessary then to fill the fuel storage tanks, erect the boiler chimney and connect up the sea-water pipes for the evaporator to be ready to produce fresh water. The pontoon, having served its purpose as a float, acts as the fresh-water storage tank. Although comparatively small, the plant has all the ancillaries of a larger plant, and is of particular interest to many arid countries where port facilities are inadequate or non-existent.

Economics

The economy of producing fresh water by the methods described must be related to the value of that 'first essential to human existence' in countries where it is lacking in the natural state.

In countries of temperate climate, with abundant rainfall, where wholesome water is produced with the minimum of treatment, it is generally so cheap that the real value is seldom appreciated. Nevertheless, progress in the production of fresh water from saline sources has reached a stage where costs are by no means beyond comparison.

The capital cost of an electrodialysis plant or a distillation plant will seldom be higher, and may be lower than that for a natural fresh-water scheme of equal capacity. The sea is all around at ground level, requiring no impounding reservoirs or deep bores, and saline waters from mine workings are in any case brought to the surface for de-watering purposes; thus no costly head works are required for initial collection.

Operating costs will vary according to local circumstances, such as in the case of electrodialysis the cost of electric current and the degree of salinity to be removed. Distillation costs will depend on the cost of fuel for steam generation, or whether waste heat is available, or bled steam from existing turbo-alternators. Although any attempt to quote exact figures might be misleading, it can be said that progress in both fields has now reached a stage where the de-salting of saline waters is well worth consideration as an alternative to conventional methods of fresh-water conservation.

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G. J. Weir Ltd., Cathcart, Glasgow.

Insulating refractory bricks

A new British Standard dealing with the classification, sampling and testing of insulating refractory bricks, has been published (B.S. 2973).

The first consideration in selecting the most appropriate insulating refractory for any application, is the limiting service temperature. The system of classification specified in this standard is based on the behaviour of the refractory material in the permanent linear change test conducted at a specified temperature. The method of determining the permanent linear change is given, and methods of determining dimensions, warpage (including convexity and concavity), bulk density, cold crushing strength, modulus of rupture and thermal conductivity are also given.

Flash Distillation Plants

By W. Smith,* A.M.I.Mech.E.

The importance of distillation as a means of producing fresh water from sea-water has been of particular importance to the oil industry—since oil wells are often situated in arid zones. A particularly efficient method of distilling sea-water is to heat it and then pass it through a series of chambers each at reduced pressure so that a quantity of steam is flashed off in each chamber. This article describes the engineering and operation of both a very large and a small package flash distillation plant.

WITHIN the last few years distillation of sea-water or brine by the flash principle has been gaining popularity and has come to be accepted, by the petroleum industry in particular, as probably the most efficient method of producing extremely large quantities of high-purity distillate. This stage has, however, only been reached at great expense and research by the few larger companies concerned with the manufacture of distillation plant. That their efforts are now beginning to pay off can be considered as only just.

A very large plant, designed to produce 1 million gal./day of fresh water was recently completed and commissioned for the Shell Cardon refinery.

The installation comprises an eight-stage flash distillation plant operating on the 'once-through' system which restricts the brine concentration to a minimum with the aim of reducing the precipitation of scale-forming salts. Contrary to general belief, the 'once-through' system is not thermodynamically less efficient than the recirculation system. The incorrect assumption frequently made is that, because a portion of the hot brine discharge from the recirculation plant is routed back to the plant as feed, instead of being discharged to waste, heat is being conserved. This is true only when the available feed is at a low temperature and the gained output ratio is high. Otherwise, the heat rejection from the plant, be it a 'once-through' design or a recirculation design, is the same; it is merely rejected from the system at a different point.

Plant details

The eight flash chambers were built in four unit sections. Starting from the higher temperature end of the plant, the first section comprises three flash stages, the second section a further three stages, and the two remaining sections one flash stage each. All the stages are arranged at ground level, as opposed to a multi-deck arrangement, so as to effect minimum loading on foundations and within the structure itself and also to facilitate ground level maintenance (see Fig. 1).

Separate vapour condensers are installed, one to each flash stage, and these follow established condenser design and constructional practices, employing a floating-head design to cater for differential expansion between the non-ferrous bundles and the carbon-steel flash chamber fabrications. The condenser surface itself comprises a multi-pass arrangement of $\frac{3}{4}$-in. outside diameter solid-drawn aluminium brass tubes, roller expanded into fixed and floating aluminium bronze tubesheets.

The plant operates with a low output ratio, that is, 4.85 lb. of distillate produced per pound of operating steam. The reasons for this are, first, that in the case of this particular project operating heat can be generated inexpensively and, secondly, low capital cost of the plant and ease of operation were important considerations. Under these circumstances the plant could be designed around a small number of stages with the advantage of greater available pressure drop between adjacent stages and a plant less susceptible to operational variations.

The pumping equipment comprises three centrifugal pumps, each with its turbine driver, taking steam at 240 p.s.i.g. as an operating medium and exhausting into the heat input section of the plant at a pressure slightly in excess of atmospheric.

Air extraction is performed by conventional two-stage steam-jet air ejectors and associated condensers, with a booster jet, exhausting to atmosphere, to facilitate rapid start-up.

Automatic controls and recorders are incorporated to fulfil the following functions:

(1) Recording the feed input rate to the plant.
(2) Recording the temperature of the feed at exit of the highest-temperature vapour condenser.
(3) Steam input controller, which combines the functions of pressure reducing from 240 to approximately 5 p.s.i.g. and, throughout control, regulated by the operating pressure in the heat input vessels.
(4) Level control in the heat input vessels to maintain a water seal between the latter vessels which operate at essentially atmospheric pressure and the first-stage flash chamber which operates at a vacuum of 7 in. Hg.
(5) Recording the distillate production.
(6) Cavitation control of the brine pump, effected by a control valve in the steam line to the pump driver, actuated by a level controller in the pump suction system.
(7) Cavitation control of the distillate pump effected by a similar arrangement to the brine pump control.
(8) Quality control of distillate output effected by conductivity meter.

Operational details

Some of the versatility of the plant can be appreciated from the different modes of operation which may be employed. Initially the plant will

*Buckley & Taylor Ltd.

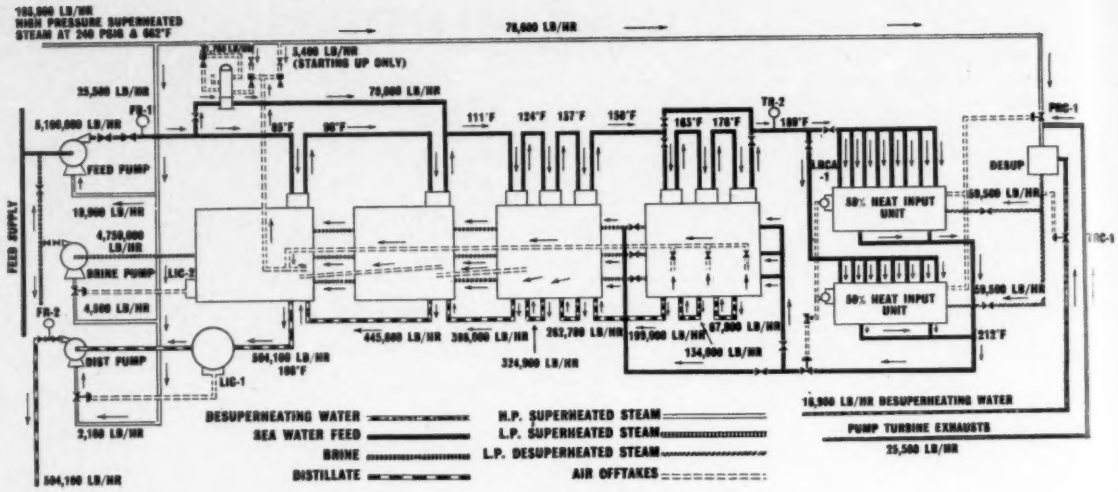


Fig. 1. Flow diagram of flash distillation plant

operate as a normal distillation plant, using as a heat source pressure-reduced and desuperheated steam from a supply at 240 p.s.i.g. and 662°F. At a later date a turbo-generator may be installed and steam at approximately 5 p.s.i.g. from the back-pressure turbines will be fed to the plant.

A distinct alternative service will be possible later when, by valving off the appropriate sections of the plant, steam from a condensing turbine set may be condensed in the plant, which in this role would function simply as a condenser. This mode of operation enables the generators to produce 50% more power if the plant is switched to this service.

The flow diagram illustrates the plant in its distilling service operation.

Design considerations

The major design problems posed by this particular project were:

(1) Sealing of the heat-transfer surfaces.
(2) Corrosion/erosion activity.
(3) The need to employ proved engineering in a new and novel design.

In addition to these obvious problems, there were a number of others of interest, connected with the sizes and weights of the unit sections of the plant to accommodate manufacturing, shipping and site erection limitations.

Scaling potential

The scaling potential is certainly the principal problem in any sea-water evaporator. In the past, and particularly in the case of a conventional submerged coil evaporator, the scaling effect has been accommodated only by building descaling routines into operational procedures, which had to be carried out very frequently to prevent undue build-up of scale on the heat-transfer surfaces of the plant.

With the advent of the flash evaporator it was at first widely acclaimed that the scale problem had at last been overcome, because the flash evaporator was considered to be scale-proof. There was some, though not complete, justification for this optimism. The flash evaporator can be designed so that only totally unconcentrated sea-water is presented to the heat-transfer surfaces. Change of phase from liquid sea-water to flash steam vapour takes place where there is no heat-transfer surface, so that only sensible heat transfer obtains in the sections of the plant where heat-transfer surface is installed. It is, therefore, impossible for any localised highly concentrated droplets of brine to deposit scale upon the heat-transfer surface.

It is true to say that the flash plant can be almost scale-free providing certain limitations are applied in the design. The principal one is to limit the maximum temperature of the sea-water, in those sections of the plant where it is adjacent to a heat-transfer surface, to a value at which it is known that almost no scale is deposited. This is a practical proposition for plants of relatively small output, but it becomes uneconomical to the degree of being impracticable in the case of large plants of, say, 1 million gal./day. output. Designs for plants of this order of size have to employ as long a flashing range as possible to avoid excessively high feed throughput requirements, so that a high maximum feed temperature is virtually mandatory.

In such a case, scale-forming salts will precipitate out of the feed and scale will deposit on the heat-transfer surface. It occurs at a slow rate compared with previous conventional designs of sea-water evaporators employing steam-heated, submerged coils, but it is still a problem which requires the utmost consideration if a large-capacity flash plant is to be a successful installation. Indeed, lack of adequate consideration being devoted to this aspect could certainly mean the difference between a trouble-free plant and a perpetual problem child.

Scale prevention

Three basic features are incorporated into the design of the Cardon plant to accommodate the scale problem. These are:

(1) Operation with minimum sea-water concentration throughout the whole plant and particularly where the feed is adjacent to a heat-transfer surface.
(2) A by-pass arrangement in the feed and brine streams around the high-temperature end of the plant.
(3) A new but proven method of introducing the external steam heat into the plant, which cannot be affected by scale deposition.

With reference to (1) the plant operates on the 'once-through' system, as opposed to the recirculation system, so that concentrated sea-water is never in contact with the scale-prone heat-transfer surfaces. The highest brine concentration which is obtained in this 'once-through' design is approximately 1.1 and this occurs only in the

final flash chambers where the brine temperature is of the order of 110°F. and the brine is not in contact with heat-transfer surface. In the condenser train, where the feed is adjacent to heat-transfer surface, the sea-water is not concentrated at all.

The by-pass arrangement referred to in (2) is installed for the specific purpose of allowing the plant to be in continuous operation, while the higher temperature condensers are valved out of stream for descaling and/or normal inspection and maintenance.

The third factor concerning the heat-input arrangement is most important. The heat-input section of a flash plant has to handle the highest-temperature sea-water which is precipitating more scale-forming salts than elsewhere in the plant. Also a given amount of scale forming on the heat-transfer surfaces of a conventional tubular heat-input unit has a much more marked effect upon the performance of the plant than the same amount of scale forming on the vapour condenser surface. If the heat-transfer surface in a shell and tube heat input was scaled to the extent of being 50% ineffective the output of the plant would be reduced by approximately 50%, whereas if the surface in the highest-temperature vapour condenser were scaled so severely as to be totally ineffective, the output would only be reduced by $1/n$, where n is the number of stages.

For the Cardon project the method which was developed to get the heat into the feed without incurring the scale problem was to employ direct injection of the heating steam into the sea-water feed, so that there is no heat-transfer surface to which the scale can adhere. The heat-input unit was designed to fulfil an additional distinct function, that of degasifying the feed with a view to purging potentially corrosive gases (principally CO_2 and oxygen from the plant) before the flash vapour is generated. This was done by introducing the sea-water into the direct-heat input unit in the form of a series of fine sprays which are heated and scrubbed by the heating steam so that the requisite heat is added to the feed whilst adequate degasification is also being carried out.

The sole disadvantage of this method of circumventing the scaling problem is that a portion of the plant output has to be made available for replacing the steam which, having been injected into the sea-water, is not recoverable. However, the advantages which are offered far outweigh this drawback. The use of this direct heat-input device in the flash distilling cycle is the subject of a patent application which was lodged following successful development work on a works test rig employing this device.

Corrosion/erosion activity

On economic considerations, the materials of construction of the large-size flash distillation plant choose themselves. The flash chamber fabrications and the vapour condenser waterboxes must be in reasonably inexpensive materials; hence carbon steel and cast iron or carbon steel are the respective obvious choices.

There are potentially few corrosion troubles in a large-scale flash plant. Firstly, by the nature of their application, large flash plants are designed to operate continuously so that the contact parts are not subjected to repeated alternatively wet and dry environment. Secondly, since adequate facilities for purging corrosive gases from the plant are conveniently accommodated in the design of a flash plant, a further important hazard is eliminated.

If the corrosion problem is to be effectively contained, however, certain precautions are advisable, particularly where dissimilar metals are in contact with one another.

In the case of the Cardon project it was decided to make use of protective coatings with two specific purposes in mind, the first being to protect the internal surfaces of the flash chambers against corrosion/erosion attack and, secondly, to ensure that the made water would not be subject to iron contamination.

In the higher-temperature flash chambers the entire internal surface is protected by a 0.030-in.-thick multi-coat solution application of neoprene. In the lower-temperature flash chambers where there are virtually no corrosive gases, it is necessary to coat only that section of the fabrications which house the condensers. Coating of these sections is carried out in all stages, its purpose being to preclude iron contamination of the made water. The heat-input units are also coated internally with neoprene in the form of a lining, in order to provide a thicker protective layer.

Cardon water has a particularly notorious reputation as a corrosive agent and, with this in mind, it was decided also to apply the solution protective coating to the condenser waterboxes and the tubesheet faces, the sea-water pipelines and the valve bodies in these lines and the distillation pipes.

Application of proved engineering

The design adopted for the Cardon plant comprises an individual vapour condenser for each flash stage. All condensers are identical and are a two-pass arrangement employing floating-head construction so that, in the event of a particular condenser failing, it can be withdrawn from the plant for repair while a common replacement condenser is installed in its place. By incorporating a floating-head design a positive tube/tubesheet joint is possible. In the case of the Cardon plant, the tubes are roller expanded into the tubesheets, but it should be noted that, if necessary, the tubes could be welded into the tubesheets.

A feature of mechanical design which required consideration was the inter-stage transport of the feed. Large throughput rates are involved and initially it was thought that, with low cost as a consideration, it was possible to utilise integrally fabricated passageways which could be built into the parent vessel fabrication.

The low-cost appeal of this arrangement, however, was considered to be more than offset by the less favourable maintenance facility. In the event of either accidental damage, or undue material wastage by corrosion/erosion activity, repair by the maintenance staff would be difficult and time consuming, when compared with replacing a corroded section of externally mounted interstage pipe. The latter is a common enough maintenance job and presents no difficulties. As a result it was decided to employ conventional flanged pipes, so that the facility for easy maintenance would be available.

The fluid-flow problem of inter-stage brine drainage required considerable design study. If this transport function was to be performed effectively, three factors had to be accommodated:

(1) The pressure drop, developed in transporting the brine between adjacent stages, must be low.

(2) The downstream terminal arrangements where mixed phase flow is developed must be such that adequate area is provided for the flashed vapour to dissociate from the residue brine.

(3) The interstage arrangement as a whole should be engineered to preclude unstable operation, when operational variations are offered to the plant.

The design which was developed for the Cardon installation employs a duct arrangement, proportioned to ensure the low velocities conducive

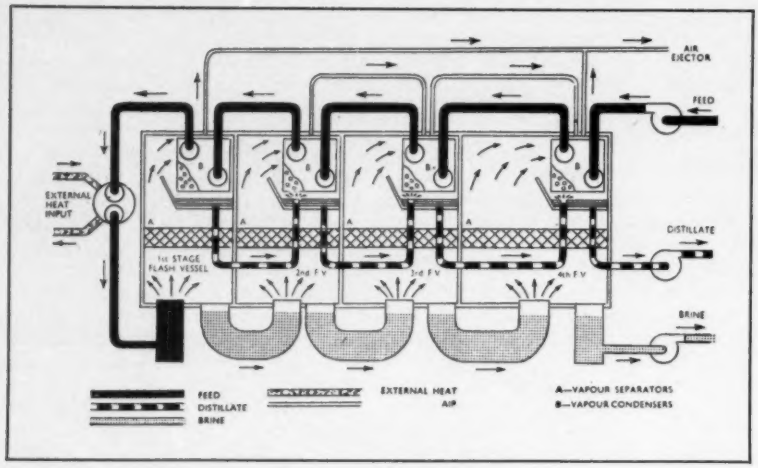


Fig. 2. 'Aquaflash' plant

to low pressure drop. These ducts take the form of a U-leg, so that a water seal between adjacent stage flash chambers is ensured at all times, regardless of operational variations, such as feed throughput changes, feed temperature variations and air ejector performance variations. At the exit end of the ducts a specially shaped impingement plate deflects the flow so as to present a maximum face area of the flashing feed, to enable the generated vapour to separate efficiently from the residue brine.

It should be appreciated that large-capacity units required to be individually engineered to suit the particular service conditions to which they will be put, so that it is not suggested that this design arrangement would necessarily be the best for all large plants.

One overriding common factor applies, however, to most large land-based sets. Since it is rarely practicable to cater for sufficiently large storage reservoirs to allow these plants to be out of service without seriously affecting the made water availability, continuous stable and trouble-free operation must be regarded as the most important design consideration.

When any plant is fulfilling such a basic function as transforming seawater into fresh water for either refinery process service, boiler feed make-up service or for domestic consumption, it must be expected that in every case where the designer has to choose between a low-cost design with potentially less reliability than a more costly alternative offering maximum reliability, the latter would invariably be employed.

'Aquaflash' plant

Just as the 1-million-gal./day plant at Cardon was nearing completion in July, the development was announced of a self-contained commercial flash distillation plant, *Aquaflash*, powered by a diesel alternator and designed to produce only 10 tons/day of distillate.

The overall dimensions of the *Aquaflash* plant are approximately 10 ft. long, 6 ft. 4 in. wide and 7 ft. 6 in. high. The sole energy input of the plant is in the form of diesel fuel to power the diesel alternator, so that it is entirely independent of an outside power supply. It can function for long periods without any operator attention whatsoever and maintenance is extremely low.

The whole equipment is arranged on two bedplates or sledges, one for the distillation plant and its pumps and the other for the power unit. These two sections need to be interconnected only with regard to electrical cables and flexible inlet and outlet pipes for the engine jacket cooler.

Basically, the operation of this plant is the same as that of the larger, permanently sited *Aquaflash* plants. Raw-water feed is circulated in series through the tubeside of four vapour condensers, its temperature being progressively raised. After leaving the highest-temperature vapour condenser it passes through the tubeside of the diesel engine jack cooler where its temperature is further increased. Additional heat is added in an electrical immersion heater vessel before it is introduced to the train of four flash chambers, arranged in series and with progressively reduced sub-atmospheric pressures.

The feed water flashes into steam in these four chambers and, in condensing in the associated vapour condensers, serves to provide heat to the incoming feed.

The condensate is withdrawn from the plant by an extraction pump which discharges it to suitable storage tanks. After the successive flashing process, the unflashed feed, which has become slightly concentrated, is normally discharged from the plant as waste. If the incoming feed is at a low temperature, however, a certain amount of recirculation can be facilitated to ensure sufficiently high plant feed temperature. But at the design point of 85°F. no recirculation is necessary.

Size reduction symposium

The Process Engineering Group of the Association of German Engineers, together with DECHEMA, is organising an European symposium on size reduction which will take place in Frankfurt (Main), Germany, from April 10 to 13, 1962.

All those engaged in this branch of chemical engineering are invited to participate in the symposium. Further details can be obtained from Verein Deutscher Ingenieure, Rheingau Allee, 25 Frankfurt (Main) 7, Germany.

Coal science conference

The next international conference on coal science, the fifth of its kind, will be the first to take place in the U.K. in 1963.

The conference, which meets every two years, is to be held at the Town Hall in Cheltenham from May 28 to 30, 1963. The first three conferences were held in Holland. Details are obtainable from Mr. R. G. J. Kingsmill, National Coal Board Coal Research Establishment, Stoke Orchard, nr. Cheltenham, Gloucester.

Dosemeters

The new British Standard B.S. 3385 lays down requirements for direct-reading personal dosemeters for *x*- and gamma radiation. It recommends that, when the dosemeter is used in the presence of beta radiation, an appropriate sleeve should be fitted to the instrument. It also points out that unsealed instruments may be seriously affected by large changes in humidity, changes in pressure and by the presence of significant concentrations of radioactive or ionised gases.

A 300,000-gal./day sea-water evaporator designed by Richardsons Westgarth & Co. Ltd., supplied to Kuwait Oil Co. Ltd.

Principles of Low-pressure Flash Evaporators

By W. Steiner,* B.Sc.(Eng.), A.M.I.Mech.E.

Very large flash evaporation plant is currently being designed and constructed for water desalination. It is therefore of prime importance to understand the fundamental principles of flash evaporation. These are discussed in this article, which in the first part analyses conditions obtained in a single flash chamber. The thermodynamics of evaporators are then discussed and various economic evaporation cycles are propounded. Finally, various problems facing designers such as feed water treatment, scale and corrosion prevention are stated.

THE traditional type of evaporator is the submerged-coil type in which a tube or tubes containing steam are submerged in water boiling it. The vapour thus produced is then condensed in a separate condenser. In order to increase the thermal efficiency, a series arrangement was adopted in which the vapour produced by boiling water was used to boil further water.

Although this arrangement increases the thermal performance of the equipment, physical limitations prevent a full exploitation of the theoretical economics available and the maximum number of submerged-coil evaporators installed in series have been six, giving approximately 5½ lb. of distillate/lb. of steam used. In addition, scale prevention in a submerged-coil evaporator is difficult owing to the high salt concentrations which may occur in the locality of steam bubbles formed in the boiling liquid.

Due to the above-mentioned limitations of submerged-coil evaporators, many people have considered the possible application of the flash principle for evaporating water. The largest user of the sea-water evaporators has probably been the Merchant Navy but, due to the development of oilfields in dry areas such as the Middle East, a demand arose for large plants for supplying drinking water for whole communities. In the case of oilfields, a supply of natural gas is available which would otherwise be wasted, so that the fuel cost of producing water tends to be low.

In more recent years it has become evident that water shortages are likely to develop in industrial countries, for instance in the United States, and, as a result, a large-scale programme of research and development into various methods of producing fresh water has been instituted. Further problems are arising due to the fact that, as the demand for water by industry increases, estuaries of rivers

**Senior Engineer, Richardsons Westgarth & Co. Ltd.*

such as the Rhine are becoming progressively more saline so that factories established there can no longer use water taken from the river.

The above facts have led in recent years to a rapid development of evaporators, particularly of the flash type, not only for drinking water purposes but also for industrial uses such as boiler make-up.

Flash principle

The idea of obtaining steam by heating water under pressure and then releasing the pressure can be shown on most thermodynamic charts, but is perhaps best illustrated on a heat-temperature chart (Fig. 1). This shows that, if water at a pressure P is heated from point O, the temperature will increase until point A is reached. Any further addition of heat will produce steam at a constant temperature until point B is reached, at which point the water will have been evaporated, and any further heating will increase the steam temperature as shown on the line BC. If the water is heated at lower pressures the same phenomena will occur but the vaporisation will take place at a lower temperature. For example, the curve for pressure P is $OA'B'C'$.

If, therefore, we heat water at a pressure P or greater to a temperature A and pass this water without the addition or loss of heat into a second vessel or compartment whose pressure is p, we end up at a point X from the diagram, thus obtaining a water-steam mixture where the percentage of steam produced or 'flashed off' is $A'X/A'B'$. It should be noted that the temperature of the steam so produced corresponds to the water temperature A' and for heat-transfer work has been considerably downgraded from the original temperature A.

In order to avoid this, the water could have been passed through a series of vessels or compartments each at reduced pressure, say $p_1p_2p_3p_4$, etc., and by extracting steam at each compartment, the path $AX_1X_2X_3X_4X_5$ is followed. By this means, substantially the same weight of steam is produced but at stepped temperature levels more useful for heat-transfer apparatus. For example, the steam produced at temperature X_2 could have heated the original water from temperature X_4 to X_3. Similarly, steam at temperature X_1 could have heated the water from X_3 to X_2, so that a large percentage of the initial heating could be achieved by use of the steam produced in the stepped flashing process.

LIST OF SYMBOLS

A	=	surface area
C_p	=	specific heat
d	=	distillate produced
H	=	heat given up by steam in heat input section
L	=	latent heat of steam
l	=	temperature loss
ΔT	=	stage temperature drop
T	=	temperature of water entering flash stage
t	=	temperature of water leaving tube system
t_s	=	saturated steam temperature
t_o	=	temperature of water entering tube system
t_f	=	temperature of water leaving last flash stage
Q	=	quantity of water
U	=	heat-transfer coefficient
Y	=	yield

Conditions in single-flash chamber

Let us now consider a typical compartment of a evaporator (Fig. 2) in which, for simplicity, we assume the same set of water to be flowing in the flash chambers as in the steam condensing system.

If the temperature of the water entering the flash chamber is T and that leaving the tube system is t and, if the stage temperature drop is ΔT, then the temperature of the steam entering the condensing section is given by:

$$t_s = T - \Delta T - l$$

where l represents the temperature loss due to the steam release and any throttling that may take place before or in the condenser section.

The effective temperature difference in the condenser is

$$\Delta t = \frac{\Delta T}{\log_e (t_s - t + \Delta T)/(t_s - t)}$$

or, taking the arithmetic mean, although less accurate, to illustrate better the parameters

$$\Delta t \simeq t_s - t + \frac{\Delta T}{2}$$

$$\simeq T - t - \frac{\Delta T}{2} - l$$

and the tube area of condensre required

$$A \simeq \frac{QC_p\Delta T}{U[T - t - (\Delta T/2) - l]}$$

(where U is the condenser heat-transfer coefficient), but since

$$\frac{QC_p\Delta T}{L}$$

is the weight of steam given off (or distillate produced), the area/lb. of distillate produced

$$\frac{A}{d} \simeq \frac{L}{U(T-t) - [(\Delta T/2) + l]}$$

or more strictly

$$\frac{A}{d} \simeq \frac{L \log_e (T - t - l)/(T - t - l) - \Delta T}{U \times T}$$

It will be seen that the area required to produce a given quantity of distillate can be decreased by increasing the terminal temperature difference $(T - t)$, by decreasing the temperature loss l, or by decreasing the stage temperature drop ΔT. The other point to be noted is that there is a minimum value of terminal difference that can be obtained even with an infinite area of condenser surface and is given by $(T - t)_{min} = \Delta T + l$.

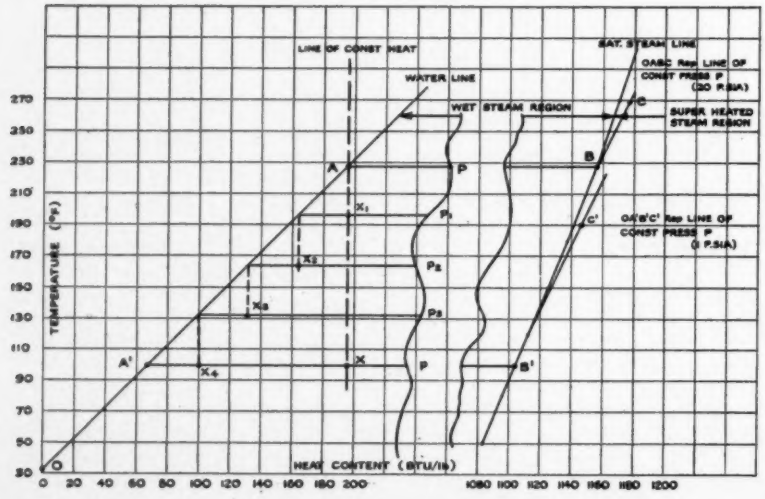


Fig. 1. Heat-temperature chart

Thermodynamics of flash evaporators

As described in the previous section it is advantageous from the thermodynamic point of view to build evaporators with several flash chambers in series. The simplest cycle is shown in Fig. 3. It will be noted that water enters at a temperature t_o and is heated in tubes to a temperature t. It then enters the heat input section where its temperature is raised to T and flashes through flash chambers, thus reducing the temperature to t_F.

It should be noted that the heat supplied to the plant from external source in the heat input section is eventually discharged to waste. Only in this way can the temperature differences necessary to operate the plant be obtained.

As shown on the diagram, the water in the flash chambers flashes in a series of steps as its pressure is reduced from chamber to chamber. Although, as described previously, the number of flash chambers used is the most important variable in the design, it is advantageous to consider the process with an infinite number of flash chambers and adjust for the final number of chambers at a later stage.

The following relationships may then be easily derived:

Mean flash chamber water flow =

$$Q - \frac{d}{2}$$

Distillate produced =

$$\left(Q - \frac{d}{2}\right) \times C_p \times \frac{(T - t_f)}{L}$$

The heating surface in the main part of the evaporator =

$$QC_p \times \frac{(T - t_o)}{U \times (T - t - l)}$$

In the heat input section the steam from an external source is used to heat the water from t to T.

$$\text{Steam used, lb./hr.} = \frac{QC_p\,(T - t)}{H}$$

where H = B.T.U./lb. given up by steam in the heat input section.

Lb. of distillate made/lb. steam used:

$$Y = C_p\left(Q - \frac{d}{2}\right)\frac{(T - t_f)}{L} \times \frac{H}{QC_p(T - t - l)}$$

With little loss of accuracy $Q - (d/2) = Q$, so that

$$Y = \frac{(T - t_f)}{(T - t - l)} \cdot \frac{H}{L}$$

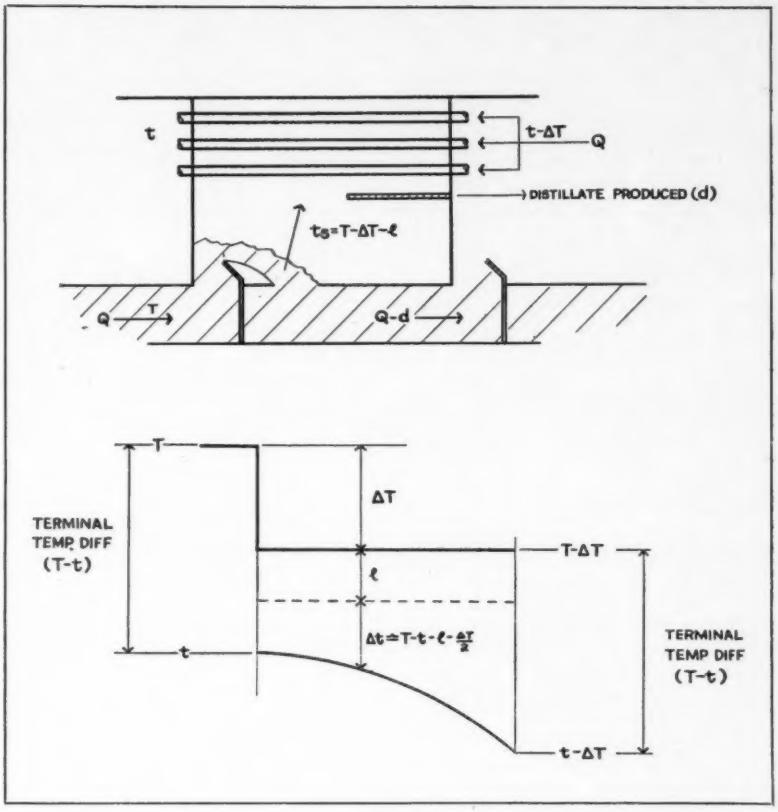


Fig. 2

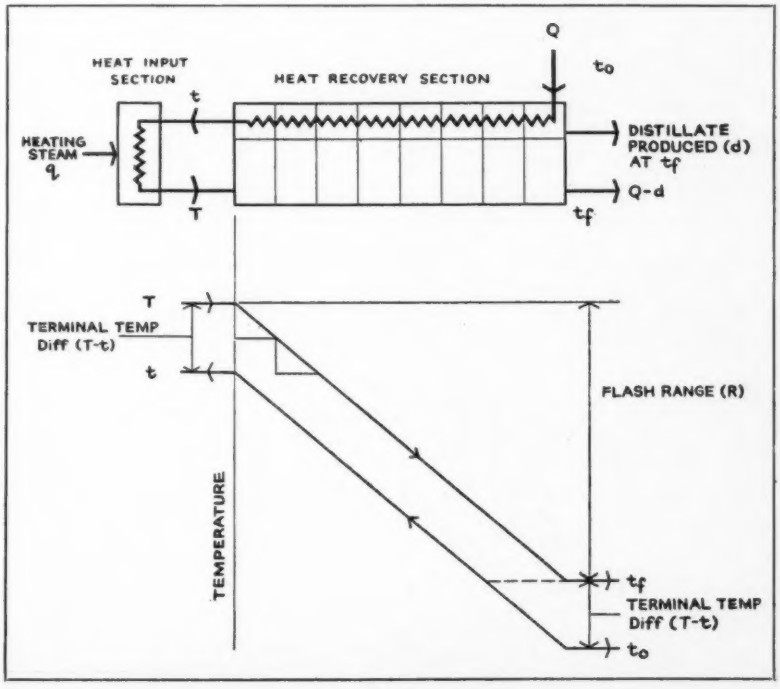


Fig. 3

In the special case where $L = H$

$$Y = \frac{(T - t_f)}{(T - t - l)}$$

$$= \frac{\text{Flash range}}{\text{Temperature difference in heat recovery section}}$$

The ratio Y is known as the ' yield ' of an evaporator, a term universally used when referring to potable water evaporators.

A better coefficient of performance is the amount of heat used to produce a lb. of water.

B.T.U./lb. distillate

$$= \frac{[Q - (d/2)]\, C_p\, [(T - t_f - l)/L]}{QC_p(T - t)}$$

making the same approximations as before

$$\text{B.T.U./lb. distillate} = \frac{(T - t_f - l)L}{(T - t)}$$

Basic evaporator cycle

The basic evaporator cycle shown in Fig. 3 is actually used in sea-water evaporation. This cycle suffers from several disadvantages:

(1) Any chemical treatment required to avoid scale deposition necessitates treating of the whole of the quantity of water entering the evaporator.

(2) Dissolved gases must be removed from the whole of the water entering the evaporator.

(3) When starting temperature $t = t_o$, the temperature of water leaving the heat input section must be raised above the saturation temperature available from the air evacuating equipment, so as to produce some flashing. This results in slow evaporator starting and can lead to troubles with high steam velocities in the heat input section, which during start-up will operate under a high vacuum.

(4) Due to seasonal variations, the value of t_o will vary throughout the year and there is no way of controlling the effect of t_o on the evaporator plant operation. This means that in winter the evaporator will, subject to adequate air evacuating equipment, operate at a much lower last-stage pressure than designed, resulting in high steam volumes which can give rise to priming and flooding of chambers. Admittedly, under these conditions it is possible to produce a much larger quantity of distillate than designed, but this is only the case if the evaporator is sized to avoid the above troubles.

Fig. 4 shows a modified cycle in which liquor is circulated rather than being introduced into the plant and

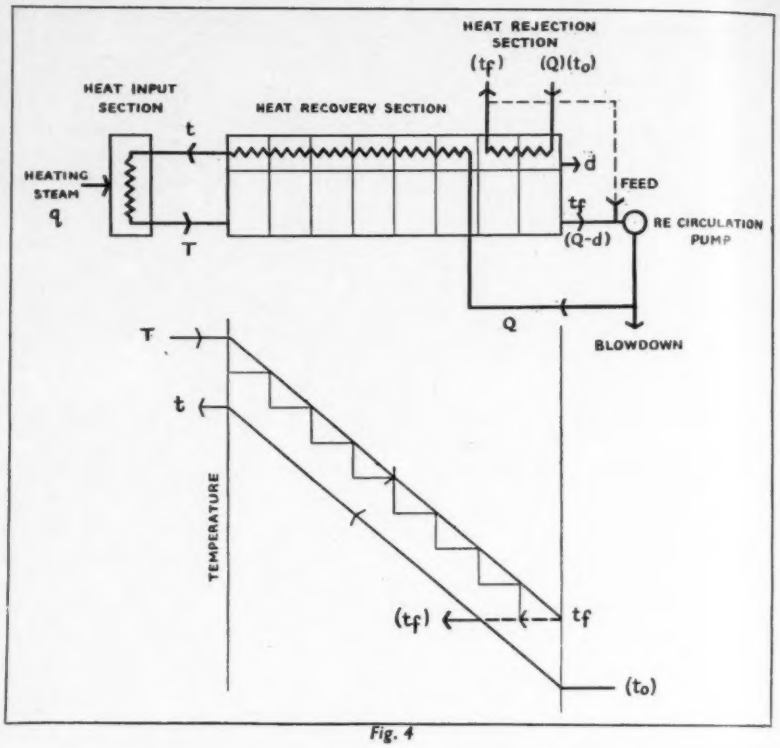


Fig. 4

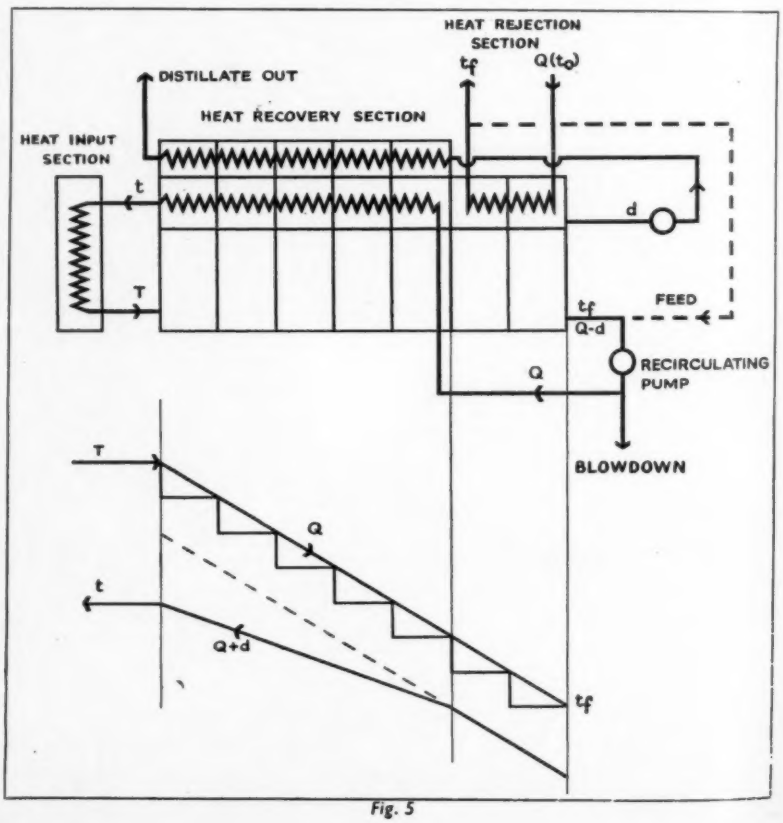


Fig. 5

then discharged. Chemical treatment is now only required for the feed to the evaporator which, in most cases, is approximately equal to twice the evaporation. Similarly, the quantities of non-condensable gases to be removed has been reduced and it is possible to control the effect of varying cooling-water temperatures by adjustment of the cooling-water quantity to the heat rejection section. This may be achieved by throttling or recirculating cooling water to the pump suction, or by the installation of heating surface in parallel rather than in series with the flow of circulating liquor.

Thermodynamics of evaporator cycles

In the case of evaporators used for the production of drinking water, the term defined as ' yield ' in the previous section is now universally used. Whilst it is adequate as a performance coefficient for potable water evaporators under defined steam conditions, it is not satisfactory when considering evaporators which produce water for process or industrial power station use.

Referring to Fig. 4 showing the recirculating evaporator circuit diagram, it is evident that the running cost of the equipment will depend upon the heat rejected to waste. In Fig. 4 there are four sources where such losses occur.

(1) The heat rejected to cooling water.
(2) The heat rejected in the blowdown.
(3) The heat rejected in the distillate produced.
(4) Radiation and other parasitic losses.

It should be noted that item (3), the heat rejected in the distillate, is, in the cycle shown, a necessary consequence of the temperature differences that are required to operate the plant and that, in the case where the water produced for the evaporator is used for drinking purposes, it is justifiable to consider the increased temperature of the distillate produced as a loss.

If the water produced by the evaporator is to be used by a process, the possibility arises that the process may require water at an elevated temperature and, therefore, in this case, this loss would not arise.

There are several possibilities available to the designer by means of which the performance of an evaporator may be increased without the capital cost of the equipment being increased and, in suitable circumstances, it is even possible to obtain an increase of performance whilst reducing the capital cost of the equipment.

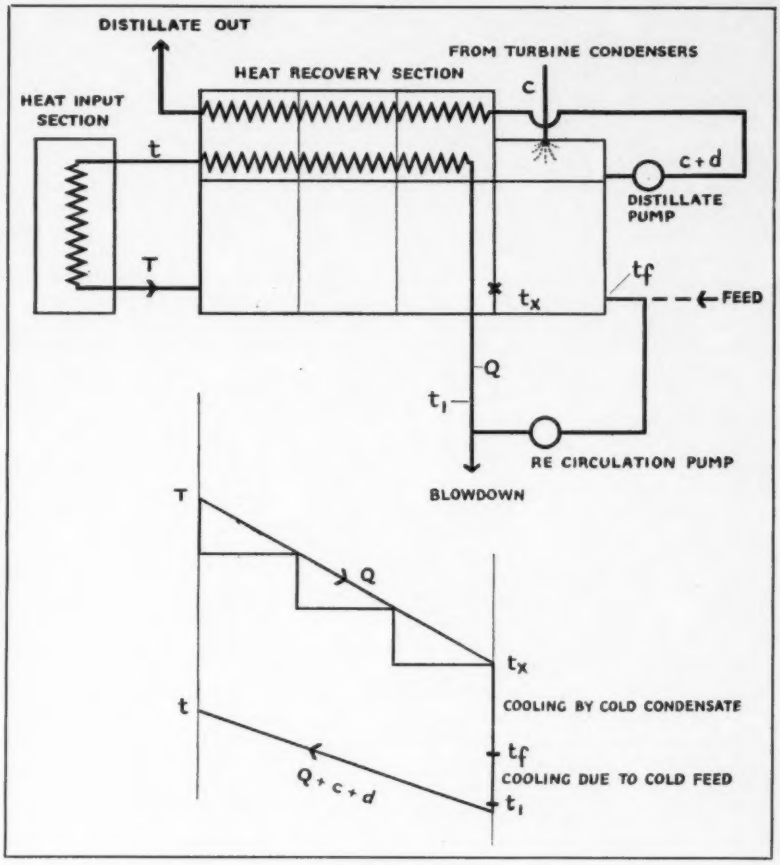


Fig. 6

For industrial uses each case must be considered individually, but some examples are given as follows:

(*a*) Modern boilers are usually designed to accept a fairly high feed temperature and in this case it is possible to pump the distillate produced by the evaporator through the heat recovery section and increase its temperature at the outlet. Such a scheme is shown in Fig. 5 and it should be noted that two consequences arise. Firstly, the feed is produced at a high temperature, which has the advantage that the evaporator is now operating as a low-pressure feed heater and it is possible to save capital cost of this item. Secondly, for a given rejection of heat, the capital cost of the evaporator itself will be lower. This is due to the fact that the temperature differences in the plant have increased as the quantity of water being heated in the heat recovery section is now larger than the quantity of water in the flash chambers. The temperature rise in the water that is being heated will, therefore, be less than the temperature drop in the flashing liquid. It will be noted that the steam consumption of the evaporator has increased as the flow Q is now raised through a larger temperature range in the heat input section.

This increased steam consumption is due to the distillate heating and does not, as far as the power plant thermal cycle is concerned, represent a loss.

It should be noted that, in cases where the purity of the distillate required for the boilers is such that it is desirable to install a demineralisation plant, this may be done at the distillate pump discharge where the temperature of the distillate will be suitable for use in resin beds. This de-ionisation unit will then act as a ' polishing ' unit, so that its running costs will be extremely low.

(*b*) In some cases, such as steel mills, it is the practice to install condensing turbo-alternators in the power station in order to generate as much electrical

power as possible, bearing in mind the steam requirement of the steel mill and the blast furnace gas available. It is then possible to arrange for the condensate from the turbine condensers to take the place of the cooling water normally used in an evaporator. This scheme is shown in Fig. 6. The condensate may be sprayed into the heat rejection section, thus achieving a saving in tubing or, alternatively, it may be led through tubes, thus avoiding any possible contamination due to priming of the evaporator. A scheme of this nature results in the replacement of the heat rejection section by an L.P. feed heater. The result is that the running costs of the evaporator have now been reduced to the heat carried away in the blow-down. This is at a low temperature and, depending on the water being evaporated, the blow-down may vary from anything from 100% to 5% of the evaporation. In any case the losses have been reduced to a very small figure indeed.

(*c*) In Fig. 4 it will be noted that the feed to the evaporator is taken from the cooling water outlet and, therefore, some cooling of the circulating liquor has occurred simply due to the fact that the feed is being heated from temperature t_o to t_f.

It is possible to make use of the temperature difference thus generated and design a plant which does not require cooling water in addition to that required to feed the evaporator. However, since normally the quantity of feed is a small proportion of the amount of cooling water used, such a plant would be expensive and will usually be considered in conjunction with schemes shown in Figs. 5 and 6.

There are two other advantages associated with schemes of this sort. Since the distillate is being produced by flashing, its oxygen content is necessarily reduced to a low figure. The boiler feed is, therefore, highly deaerated. Secondly, as a final protective measure, it is possible at a very low capital cost to combine the heat input section with a deaerator into which all the water going to the boiler is introduced. This provides also a convenient way of reintroducing hot streams of H.P. heater and trap drains.

In order to achieve satisfactory results with industrial evaporators it is necessary to consider their introduction into a cycle at the earliest possible moment and to obtain reliable heat and flow balances for the scheme being considered.

The output from the evaporator can be conveniently regulated by the quantity of circulating liquor circulated, but it will also be affected by external conditions, such as the temperature and quantity of cold condensate available at any particular time. It is, therefore, necessary to design for a realistic condition so as to avoid embarrassment under special methods of operation.

In the design of any particular flash evaporator, several variables are available to the designer which will affect the capital cost of the equipment, assuming that the required performance has been determined.

(1) Top temperature

The top temperature is largely determined by the characteristics of the water to be purified and the location of plant. There are three methods at present available to limit calcium and magnesium scale formation at the higher temperatures.

(*a*) *Hagevap.* This is a polyphosphate which prevents the formation of scale due to calcium carbonate. It is available in the form of a powder and is, therefore, readily shipped to remote sites. The maximum temperature which can be used with this material is in the region of 190°F.

(*b*) *Sulphuric or hydrochloric acid.* These acids will reduce the alkalinity of the feed water to an extent where scale deposits will not occur. The upper limit where it is effective has not been fully explored, but it is certainly effective up to 250°F. where constructional conditions begin to be important, due to equivalent saturation pressures. The treatment may be used both with sea or industrial waters. Acid costs are most dependent on shipping costs so that in underdeveloped countries, for instance, acid is prohibitive in price.

(*c*) *Basic exchange equipment.* Suitable for softening industrial waters, but scale deposit may occur, due to hardness leakage. A safe top temperature of 210°F. is suggested. This method is not suitable for sea-water evaporators.

Sulphate scale must be avoided at all costs as it is extremely difficult to remove. This is usually achieved by limiting the concentration of circulating liquor in order to avoid approaching the solubility of calcium sulphate scale. It should be noted that calcium sulphate exhibits a reversed temperature-solubility relationship and that this may in the future be a limiting factor.

(2) Last-stage flash chamber temperature

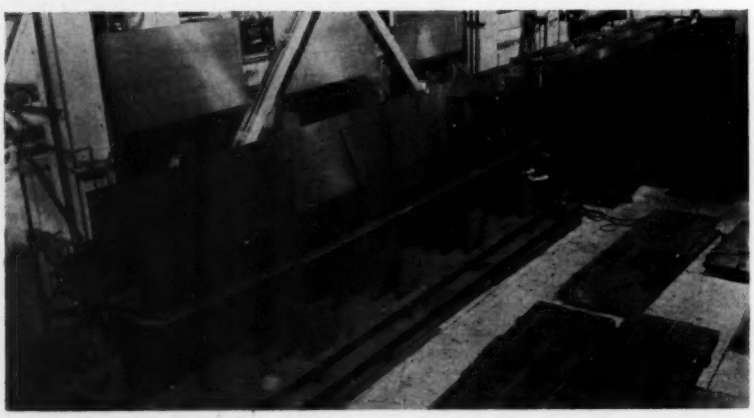
The bottom temperature is determined largely by the climatic conditions and it will be appreciated that the lower the lowest circulating liquor temperature, the larger the quantity of cooling water which has to be supplied to the heat rejection section.

In industrial evaporators, in which the temperature differences required for the operation of the plant are obtained, by cooling by feed and/or feed heating of cooling condensate, there will be an optimum lower temperature.

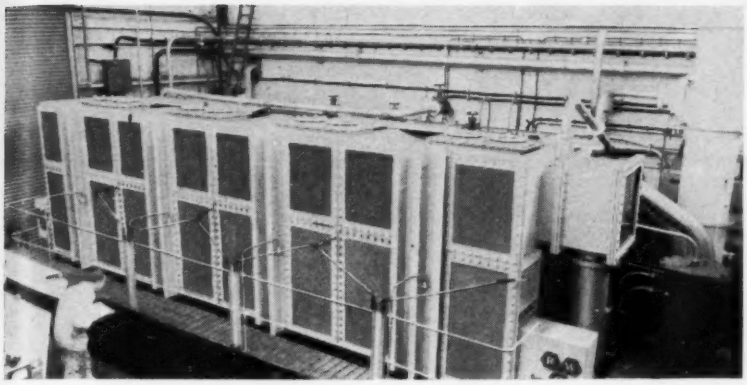
(3) Number of flash stages

As shown in previous sections, the number of flash stages is of great importance, due to its influence on the heating surface required. Increasing the number of stages will increase the physical dimensions of the evaporator as well as increasing the cost of the vessel. There is, therefore, an economic number of stages where any particular design at the point where the increase in vessel cost is balanced by the saving of heating surface.

In some designs there will be a



A 150,000-gal./day Richardsons Westgarth vacuum flash evaporator under construction



A Richardsons Westgarth multi-stage vacuum flash evaporator test rig

limiting number of stages, due to the difficulty of transferring the circulating liquor from stage to stage with the low-pressure differentials available.

(4) Tube diameter

The tube diameter has an optimum value as with the increase in tube diameter, the number of tubes is reduced and, therefore, the amount of labour used in installing tubes is reduced. Its predominant effect, however, is that an increase of tube diameter will have a tendency to increase the physical dimensions of the vessel, the tube length installed varying roughly as the tube diameter. The result is that there is frequently a limit on the diameter which can be used with a given design, due to shipping limitations.

(5) Heat-transfer coefficient

The heat-transfer coefficient from steam to water has been thoroughly explored in the past and there is no difficulty in obtaining realistic data from the literature on the subject. Some increase in the value of the heat-transfer coefficient is available by increasing the tube velocity. There is, however, a severe penalty due to increased pump powers required.

Evaporator design

In an evaporator design a number of problems must be overcome to achieve satisfactory performance.

(*a*) It is of vital importance to eliminate or limit scale formation. This is usually achieved by suitable limitation of the top temperature and suitable chemical treatment. In cases where calcium and magnesium scales occur through some cause or other, it is possible to remove these by acid washing. This has now been reduced to a routine operation and requires no more than two hours out of service for the evaporator.

(*b*) The temperature differences and the result in pressure differences between flash chambers in a modern plant of efficient design tend to be extremely low. In order to obtain a full flash-off in each chamber, it is necessary that each particle of water be brought to the surface, as the effect of static water levels can be very deleterious. It is, therefore, necessary to design suitable orifices and baffles which will achieve the above objects, as well as provide a fair degree of flexibility so as to avoid either flooding or passage of steam from chamber to chamber when the evaporator is not running under design conditions.

(*c*) In order to achieve satisfactory purity of distillate, the vapour release areas available in each chamber must be adequate. Distillate purity is usually increased by the installation of suitable separators and present-day designs are available which will give a purity of 1 p.p.m.

(*d*) The feed water entering the evaporator is usually saturated with oxygen and the breakdown of carbonates will add further non-condensables. It is, therefore, necessary to remove these in an adequate manner and in this connection it should be noted that one of the most frequent causes of unsatisfactory operation is inadequate venting. It is by no means an easy matter to achieve satisfactory venting under the low-pressure differences available and the large spaces to be vented.

Bad venting not only results in a decreased performance, due to a blanketing of tubes, but can also affect the pressures inside individual flash chambers to a degree which results in flooding.

(*e*) There are two sources of corrosion, apart from the general corrosion due to water, which can be troublesome in evaporators.

(i) Water boxes are usually constructed in mild steel, whereas the tube plates are in naval brass. As a result, electrolytic action can occur between these materials and the author's firm has now made it a practice to rubber line all water boxes. This has been done in preference to evolving an electrolytic protection system, as at the temperatures ruling in evaporators, the polarity of anodes can reverse.

(ii) Corrosion can also occur at the points where non-condensables are giving off. In this case, corrosion can be reduced to a minimum using non-ferrous construction.

Reinforced plastics in transport

A special feature dealing with reinforced plastics in transport appears in the September issue of **Fibres and Plastics.** Other articles include Petrochemicals and Plastics in Japan and Belgium's Plastics Industry.

The following articles appearing in our associate journals may be of interest to readers of CPE:

Manufacturing Chemist—Dust and Fume Control, by D. R. Dickinson. Small-scale Equipment: Filters, by B. W. Burt.

Public Works and Muck Shifter—The Application of Hydraulics to Earth-moving Equipment. The Replacement of Rollers by Balls in Excavator Slewing Rings, by H. T. Lloyd.

Paint Manufacture — Durability Characteristics of Organic Finishes, by S. K. Bose and S. N. Mukerji.

Petroleum—The Oil Rivalry in India, by Dr. J. Dubashi. Non-destructive Testing of Welded Tubing.

Food Manufacture—Editorial review of materials, methods and new packs.

World Crops—From Man Power to Machines, 2—Methods of Organising the Mechanisation of Small Farms, by J. C. Hawkins. Aerial Farming in Australia, by L. T. Sardone.

Specimen copies of these journals and subscription forms are available from the Circulation Manager, Leonard Hill House, Eden Street, London, N.W.1.

Safety Assessments in the Nuclear Industry

Part 1

By A. Quinton,* M.Sc., F.Inst.P.

More factors must govern safety assessment in chemical plant associated with the nuclear industry than are generally associated with the siting of normal chemical plant. Various such safety criteria are discussed in this article. It is pointed out that the selection of site position is affected by the position of existing facilities of particular importance to radioactive material processing; in addition there is also a need to ensure that the introduction of a new process does not produce an unacceptable hazard to surrounding buildings. References are made throughout to official recommendations and regulations which must be followed meticulously.

THE purpose of this article is to consider those additional hazards in the working of chemical plant which arise from the handling and processing of radioactive material and to state the main principles of safety assessment of such plant. These principles are mainly concerned with health physics aspects. Some of these hazards arise directly from radioactivity, but others are due to the fact that some of the reagents necessary in processing atomic energy materials are themselves dangerous. There is a further hazard if the plant is processing fissile material such as uranium enriched in the U^{235} isotope. If there is a sufficient quantity of such material assembled in the right shape and concentration a self-sustaining chain reaction may be possible and a criticality excursion may take place. Attention is drawn, as appropriate, to the possibility of mechanical damage to plant or building, the initiation of fire, the production of external radiation and contamination, but the article does not attempt to deal fully with this hazard.

The design and operation of the plant must be such that the workers themselves and the public generally are safe both from external radiation hazard and contamination. The latter is most likely associated with a fire where the radioactive vapour and dust produced spreads beyond the site boundaries in a downwind direction, but it could arise from a criticality incident.

The hazard presented by a radioelement depends on its physical half-life, its particulate size if in the form of a dust, and the part it plays in human metabolism. Radio-iodine is of particular importance due to its volatility and the fact that when it is inhaled or ingested it ultimately concentrates in the thyroid gland. The International Commission on Radiological Protection[1] has published recommendations applicable to radiation workers and they indicate the levels of concentration in air and water which would be likely to give the maximum permissible body burden in the critical organ. The recommendations take into account all those factors which are of importance between breathing contaminated air or drinking contaminated water and the subsequent radiation dose to the body. The levels appropriate to members of the public or those not classified as radiation workers are lower than the levels for occupational exposure.[1, 2]

Although this article stresses radiological hazards and possible emergencies, it is nevertheless true that the major design effort is still to achieve industrial safety as normally understood. Accidents leading to site or environmental damage are a rare occurrence and have been planned against in a well-designed plant.

Site selection

The main principle in safety assessment of a proposed site is to ensure the safety of the neighbouring population from toxic and radiological hazards including those which arise from solid waste, gaseous and liquid effluent.

At an early stage in the design of chemical plant it is usually possible to specify the scale of operations, the reagents to be used, the products of the plant and the waste materials. The plant assessor can estimate from preliminary flowsheets and flow diagrams for various stages in the working of the plant

(1) The total quantity of material at hazard.
(2) The waste products arising.
(3) The products which have to be recovered and re-used.
(4) The quantities of materials in store.

Knowing the physical and chemical nature of the materials, the form of containment or the method of storage, the assessor can estimate the total hazard associated with the chemical plant.

From a health physics point of view it is usually sufficient at this stage to concentrate attention on a few radioelements which, in the event of an emergency release, are known to be of particular hazard to the public. These include iodine, strontium and caesium, as well as the original radioactive materials uranium and plutonium. It is possible to estimate the range of hazard and the numbers of people likely to be involved and whether these numbers are sufficiently few to be acceptable.

A major chemical process may need

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both an effluent treatment plant and a pipeline to carry large volumes of very low active waste to sea. It is likely in selecting a site that one would give preference to one where the dilution of waste materials in the sea had already been investigated and proved adequate (see Fig. 1).

Smaller chemical processing plant may be such that the low active waste can be treated and delivered to a river. Since the water may be subsequently used for drinking or industrial use, it is important that the design makes provision for the monitoring of effluent and for hold-up tanks so that national recommendations[3] are not infringed. For small chemical plant, an extremely low active effluent may be discharged to the ordinary main drainage system.[3, 4] Although radioactive limitations are usually more exacting than chemical toxicities, the requirements of river boards and local authorities should not be infringed.

If the first study demonstrates undesirable features of the proposed site and there is no better alternative site available, the designers may be asked to introduce modifications to make the site acceptable. These modifications might include more effective treatment plant, higher ventilation stacks, longer sea pipelines, better containment of the plant or a reduction in throughput. One site may be found preferable to another because of a better local transport system which would enable help to be brought in quickly in time of emergency.

Position on site

The main principle in the assessment of the suitability of the position on site is to ensure the safety of the workers on the site and to avoid mutual hazards between buildings.

In order to select the most suitable area for the erection of chemical plant, it is good practice to note down on a site plan two or more areas which are available and which already fulfil the straight engineering requirements, and then to consider the following safety aspects:

(1) The numbers of workers in adjacent buildings and the public population pattern close to the site boundary.

(2) The relation of existing buildings and ventilation stacks to the proposed new buildings. (The air flow near the area should be such that the effluents can get away satisfactorily. It is only in rare cases that the direction of the prevailing wind is a major consideration.)

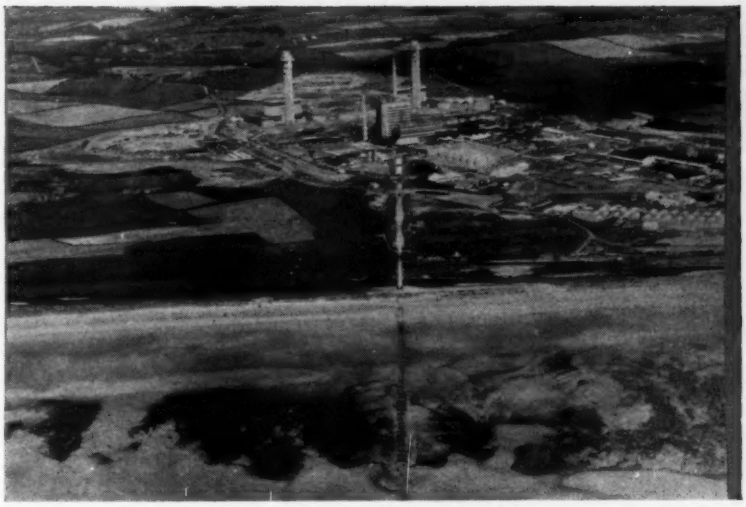

Fig. 1. An effluent pipeline at Windscale

(3) The process carried on in existing buildings to assess how, in an emergency, the proposed building will be affected and *vice-versa*.

(4) The relative position to those facilities of particular importance to radioactive processing plant, *e.g.* decontamination centre, instruments section, treatment plant, laundry, change-rooms, etc.

(5) The accessibility of existing emergency services, *e.g.* police, fire-fighting, medical care, site emergency control and emergency assembly points.

(6) The nearness of storage facilities for products wanted in the process and for storage prior to disposal.

It is unlikely that any particular area will be an ideal solution to all the problems involved. It is essential that the proposed building should not introduce an unacceptable hazard to existing plant.

Building construction

The building should be of minimum fire hazard and there should be adequate provision for emergency evacuation. There should be adequate space for health physics services. The safety of workers in the process must be fully safeguarded by adequate shielding and measures to avoid the contamination of surfaces and air.

The building should be constructed of fire-resistant materials such as brick, concrete, steel, etc. It is best to keep combustible materials such as wood to a minimum and, where their use is unavoidable, they should be treated with fire-retardant preparations. Where paint is used it should be of a quality appropriate to the area of plant, being resistant to acids, alkalis and detergents as is necessary. There are paints available which are resistant both to radiation and contamination and, in certain cases, they are also resistant to abrasion and high temperatures. It is important that the best possible use of these paints is made, as they are often superior to normal industrial paints. The use of electric cables cannot be avoided, but they are normally combustible and their location should be kept as far away as possible from a fire hazard. The layout of the building should conform to the relevant Factory Acts and it can be noted that the very provision of adequate space for plant, men and services is a help from a safety point of view.

At an early stage in the assessment a detailed plan of the building is not normally available but the designer should be aware of certain basic principles which need to be carried out during the various stages of the design. In discussing layout, it is convenient to think in terms of operational, maintenance and containment areas, and these can be fairly readily defined in production work. It is not so easy to do this if the plant is of a research or development nature, calling for greater flexibility. Entrance to the containment area—those sections of plant where the radiation and/or contamination levels are high—is restricted and men are not allowed to enter until a check on the radiation level has been made, and they would also be required to wear protective

clothing. The design should include measures to shield other areas from the containment area, and also to prevent the spread of contamination. The operational area can normally be entered without restriction and without the need to wear protective clothing, although it is not unusual for men to wear laboratory coats or overalls in this area. They can control the plant from this area without exposing themselves to levels of radiation greater than those specified in Table 1 and in Tables 2 and 3 to be given in Part II. Modifications to the plant or routine maintenance are done from the maintenance area. The men would normally wear protective clothing whilst maintenance work is being done, but for the greater part of the time the radiation and contamination levels are low and not very different from those in the operational area. The details of shielding and contamination are considered later in this paper, but at an early stage it is important that items of plant are arranged in correct positions. As an illustration of this, if the working of the plant requires sampling at various stages, convenient positions must be provided for this work to be carried out in a safe manner. The flow of ventilating air should always be away from a clean area. The total volume and the rate of flow is dependent on the type of area and the nature of the hazardous materials, but in both cases it should be sufficient to prevent the spread of contamination.

In many chemical industries, there is a need for the workers to wear protective clothing, but rarely on the same scale as is necessary in nuclear industries where the provision of change-rooms and specially designed protective clothing is an important feature. Change-rooms, in addition to being adequate to meet probable future requirements, should meet the following points:

(1) They should be so situated that they facilitate the movement of men to and from contaminated areas.
(2) Ventilation should prevent back diffusion of contamination.
(3) They should be fitted with adequate and properly positioned toilets, wash-basins, showers, and clothes cabinets, so that the men would be encouraged to use them effectively.
(4) They should be designed so that men have a minimum of walking; no area should be traversed by both clean and contaminated men.
(5) The proposed materials of construction should provide for easy decontamination, particularly on the process side of the change-room barrier (the surfaces should be hard, smooth and impermeable).
(6) The design should include the provision of radiation monitoring equipment to ensure that each individual worker leaves the change-room free from contamination.

Table 1. Maximum permissible organ and tissue doses

Exposed part	*I.C.R.P.* (*max.*) (*rem*)
(*a*) Whole body, blood-forming organs, gonads, eye lens	(*b*) 5 (N-18)
	3 rem/13 wk.
Skin, thyroid, bone	30 rem/yr.
	8 rem/13 wk.
Other organs	15 rem/yr.
	4 rem/13 wk.
Hands, forearms, feet, ankles	75 rem/yr.
	20 rem/13 wk.

(*a*) Normally means the trunk not necessarily including the extremities.
(*b*) In the table the formula $D = 5(N-18)$ gives the maximum permissible value of the cumulative dose where N is expressed in years.

Although problems of shielding and contamination are discussed in detail in Part 2 of this article, some comments are made here concerning the general design of the building. The measures taken against external radiation and contamination hazards should not be over-elaborate. It is not always necessary to design for complete containment of an unsealed radioactive material. Effective control can often be achieved by carefully placed extraction points or by ventilation hoods. For a highly hazardous material such as plutonium, particularly if the production of a fine dust cannot be excluded, such relatively simple methods would not suffice and it would then be necessary to carry out the process or the handling within a glove box (see Fig. 2).

The glove box containment ensures that the operator is protected from the contaminated atmosphere within the glove box. He is able to view the work through transparent panels and to manipulate apparatus through sealed glove ports. Whilst glove boxes do offer some protection against the softer radiations, their main purpose is one of containment. The air from extract points, ventilation hoods and glove boxes should be filtered, and the design should provide for these filters and also for their being changed from time to time.

The main emphasis in the design is to prevent contamination, but in practice there is usually some unavoidable contamination and the design therefore should facilitate decontamination by avoiding unnecessary corners, horizontal surfaces and surfaces which are not given a smooth and impermeable finish.

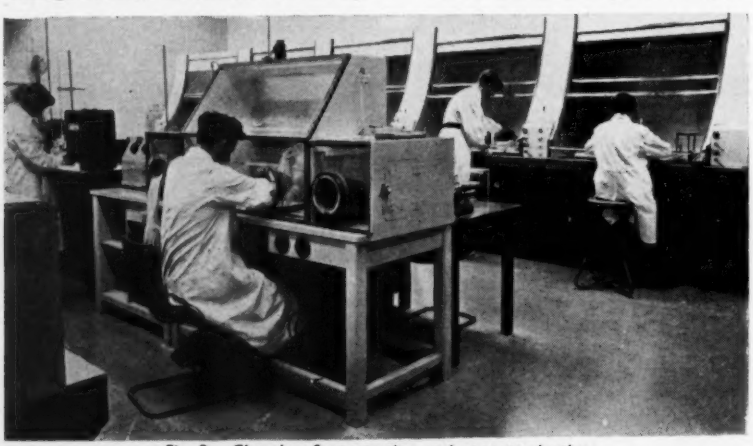

Fig. 2. Glove box for protection against contamination

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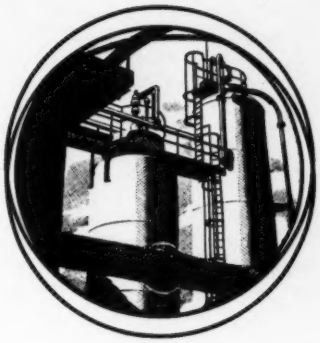
Materials of Construction for Chemical Plant



CERAMICS

By D. A. Holdridge,* B.Sc., A.R.C.S., D.I.C., F.R.I.C, F.I.Ceram.

This article describing the properties and applications of ceramics as materials of construction for chemical plant, is the seventeenth in this series. Previous articles have dealt with PVC, lead, nickel, stainless steels, graphite, polyolefines, copper, timber, platinum, titanium, aluminium, reinforced plastics, cast iron, mild steel, silver and glass. The resistance of ceramic materials to almost all chemicals, combined with the low cost of fabricated ceramic plant, make this one of the most valuable materials at the disposal of the chemical plant constructor.

IN considering the application of ceramics to the construction of chemical plant, one is faced with the problem of limiting the scope of the subject to such reactions as proceed at ambient or moderately elevated temperatures. One must exclude the metallurgical, glass and carbonising industries in which ceramics, in the form of refractory materials, are indispensable and confine the consideration to those chemical reactions in which ceramics offer one of several possible alternatives.

Even when the field is deliberately restricted in this manner, it must be remembered that the term 'ceramics' covers an extensive field of materials of differing composition, the range of which is being continuously extended to provide products with properties tailored to the requirements of the processes for which they are designed. Nevertheless, when ceramics are considered in relation to chemical plant, the mind inevitably turns to chemical stoneware, possibly to chemical porcelain or perhaps to acid-proof bodies and tiles and, when filtration problems arise, to porous ceramics. The main theme of this article will, therefore, be concerned with these products.

Historical

Vitrified stoneware, though known to the Chinese for many centuries, seems to have developed in the Western World in the Rhineland and Westerwald areas of Germany about the 12th century A.D. At the time it was chiefly confined to domestic articles, notably jugs and beer mugs. A vogue for this ware gradually developed in this country and large quantities were imported during the 16th and 17th centuries. The first known English imitator of this Continental type of stoneware was John Dwight, who started manufacture at Fulham in the later half of the 17th century.

According to Singer,[1] chemical stoneware, as distinct from the more generally domestic and art ware, was first made in the middle of the 16th century, and the same author[2] states that the retorts, used in the production of sulphuric acid by Jan D. Starck at the beginning of the 18th century at Brasy, near Pilsen, were made from a stoneware body. Nielson and Garrow[3] claim that stoneware plant was first used by the chemical industry in this country and was later taken up by the Germans, who imported clays from England for its manufacture.

[Courtesy: Royal Doulton Potteries

Fig. 1. Salt-glazed stoneware jars in use after 100 years' service

Certainly Doulton began the manufacture of stoneware at Lambeth in 1815 and, with the development of the chemical industry and the ever-increasing demand for stoneware drain pipes, were obliged to build a supplementary plant at Erith and also to acquire further manufacturing facilities at Tamworth for the same purpose. Salt-glazed stoneware jars, made 100 years ago at Lambeth and still in use,

*The British Ceramic Research Assoc.

are illustrated in Fig. 1. They should be compared with the modern, white-glazed stoneware in the background of the picture. The other well-known English chemical stoneware manufacturing company, Hathernware, with a foundation dating back to 1775, began making chemical stoneware in 1916.

In Germany and neighbouring areas on the Continent and in the U.S.A., the chemical stoneware industry appears to have developed about the middle of the 19th century, two areas involved being Aussig in Czechoslovakia and Frechen near Cologne. Perhaps the most famous Continental plant is the Deutsche Steinzeugwarenfabrik at Friedrichsfeld, between Mannheim and Heidelberg and founded about 1870, who shared with the Deutsche Ton-und Steinzeugwerke A.G., whose factory is now in Polish controlled territory, the reputation for producing the best chemical stoneware in Germany.

In a similar manner, the chemical porcelain industry is of German origin and development from the table and art ware industry founded by Böttger at Meissen about 1709. Porcelain, a white translucent material is, in effect, stoneware prepared from refined materials and matured at a somewhat higher firing temperature. The body is much less plastic than stoneware in the unfired state and is hence not as amenable to the manufacture of the larger types of article. Much of the ware from this body is hence of laboratory scale, though some larger articles are made. Famous names in the production of chemical porcelain are the Berlin State Porcelain Factory and Haldenwanger in Germany, Royal Worcester and Royal Doulton in this country and Coors in the U.S.A.

The rougher products called acid-proof bricks and tiles appear to have developed from the engineering type red and blue bricks, probably with later refinement regarding the source and type of clay used and the firing conditions to which the ware was subjected.

Composition and properties

Most of the ceramics used in chemical plant are complex aluminosilicates and are made from some form of clay either with or without the addition of other ingredients such as quartz or grog (precalcined clay) and a flux such as felspar. Articles may be formed either by plastic or slip casting processes and, after careful and complete drying, they are fired to a high temperature (1,200° to 1,300°C. for chemical stoneware and 1,400°C. for porcelain). This process converts them to hard, dense and inert products. It further proffers an indication of how the properties of chemical stoneware may be altered to suit particular conditions; any oxide, nitride or other compound that can withstand the high temperature of firing without appreciable reaction with the main ingredients can be incorporated in special resistant ceramics, often with appreciable tolerance to thermal shock. Alumina and silicon carbide have been used in this way with considerable success in reducing sensitivity to temperature gradients—one of the principal drawbacks to the use of ceramic materials on chemical plant. This aspect will be considered more fully later.

Chemical porcelain is impermeable and translucent in thin section; usually the body is white and ware is coated with a transparent felspathic glaze that matures with the body during the high-temperature (1,400°C.) firing. Chemical stoneware, on the other hand, is impermeable but opaque to transmitted light, the body is coarser and has a conchoidal fracture. In general it tends to be stronger mechanically, because it is normally used in thicker-sectioned articles, although intrinsically the porcelain body is the stronger. The physical properties of typical chemical stoneware and of porcelain are compared in Table 1.[4]

Acid-resisting bricks are usually of either the blue or red engineering types produced from a relatively coarse untreated surface clay by mass-pro-

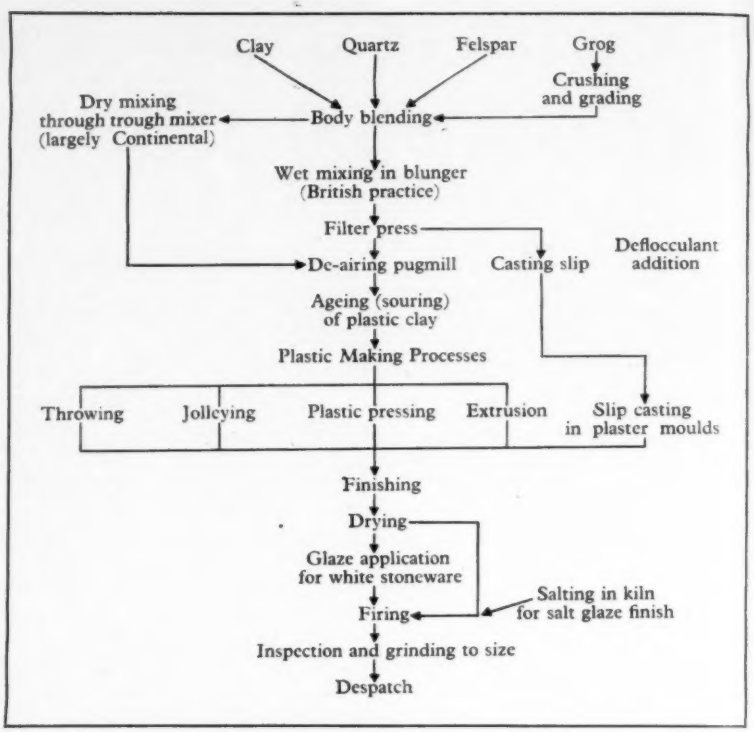


Fig. 2. Flowsheet for chemical stoneware

[Courtesy: Hathernware

Fig. 3. Making the plaster mould for a large stoneware tee-piece

[Courtesy: Hathernware

Fig. 4. Removing plaster core from a cast reaction vessel

duction methods. For more exacting conditions, special acid-resisting bricks or tiles are made from prepared stoneware bodies virtually free from iron-bearing impurities. Porous ceramics, by contrast, differ from the preceding classes in that the basis is usually a highly-siliceous material, carefully graded according to size and bonded by limited amounts of flux.

Manufacture of chemical stoneware

Body preparation may be either by the dry or wet mixing methods; the second is the normal process in this country, whereas Continental practice favours the former. A flowsheet embracing both methods is shown in Fig. 2.

In either method the body is passed through a pugmill, frequently of the de-airing type, and the issuing column of clay is cut off into convenient lengths for storage. These 'slugs' are allowed to age or 'sour' for a suitable period, this process allowing the material to even out moisture differences and, further, promoting some bacterial growth. Both these reactions have beneficial effects on the plasticity of the body. One advantage of the wet mixing process that is not afforded by dry mixing is that the body suspension ('slip') can be passed through fine sieves to extract impurities and, moreover, the issuing slip can be passed over electro-magnets to remove any magnetic iron compounds, a process that improves the appearance of the finished ware.

For plastic making, the soured body can be pressed into plaster moulds for the manufacture of square or rectangular tanks, etc., or it can be either thrown or else jolleyed into plaster moulds, if articles of circular cross-section are required, or it can be extruded when pipes are being made.

For casting, the body is converted into a slip of creamy consistency by the addition of the requisite amounts of water and deflocculants. The slip is then poured into plaster of paris moulds, the size of which allows for the subsequent shrinkage of the article during drying and firing and which is of the order of 10 to 12%. Fig. 3 shows mould makers at work on a plaster mould for a large tee-piece, Fig. 4 the removal of the core from a round-bottomed reaction vessel cast, Fig. 5 the mould being stripped from a storage jar, and Fig. 6 a tower section being finished in clay, prior to firing.

The illustrations indicate clearly that many of the articles made are large and that their thickness will, therefore, be considerable. Slow and careful drying are consequently imperative if cracks and strains in the structure are to be avoided. Clearly, smaller articles will require a correspondingly shorter drying period.

Treatment after drying depends on (*a*) whether the ware is porcelain or stoneware and (*b*) if stoneware, the mode of glazing that is to be adopted. If the ware is porcelain, it receives a prefiring to 900° to 950°C. and, after cooling, a felspathic glaze is applied by dipping or spraying, and body and glaze are then matured together in the glost firing, which is carried out at 1,400°C. Stoneware, on the other hand, may receive one of two treatments. The better-class ware is painted or sprayed with a glaze coating after drying, and body and glaze mature together during firing which, in this case, will be somewhere in the range 1,200° to 1,300°C., according to the body composition.

[Courtesy: Royal Doulton Potteries

Fig. 5. Removing plaster mould from cast storage jar

Cheaper ware is salt-glazed, a process involving firing the dried ware and feeding common salt to the fire-grates at a temperature in the range 1,100° to 1,200°C. The salt vaporises and forms fusible silicates with the body of the ware.

Table I. Physical properties of chemical stoneware and porcelain[4]

	Chemical stoneware	*Hard-paste porcelain*
Specific gravity	2.55	2.40
Porosity (%), *i.e.* volume pore space/unit volume	0.3	Nil
Tensile strength, p.s.i.	2,400	4,000
Compressive strength, p.s.i.	25,000	60,000
Modulus of rupture, p.s.i.	5,000	10,000
Linear coefficient of expansion (20° to 100°C.)	4×10^{-6}	4×10^{-6}
Thermal conductivity (K cal./sec.cm.°C., 20° to 300°C.)	0.0015	0.0014
Specific heat (K cal./kg.°C., 20° to 100°C.)	0.19	0.20

Firing of large pieces takes place in intermittent kilns, but smaller pieces are sometimes fired in continuous kilns. Normally the ware is heated slowly in the early stages; adsorbed water is removed up to about 250°C. and the clay molecule decomposes at about 550° to 600°C., the OH groups being eliminated as water. At 573°C., any α-quartz inverts to β-quartz, the process involving an expansion of the order of 1%, and up to 700°C. any carbonaceous matter associated with the body is burned out. The result of these reactions is that the body is now mechanically in its weakest state; a considerable weight loss has occurred and virtually no vitrification has taken place. Beyond 900°C. incipient vitrification begins with the naturally occurring fluxes in the clays and the reaction proceeds with increasing vigour as the temperature is raised and the added fluxes begin to act.

When the clay mineral, which is essentially kaolinite, decomposes at 550° to 660°C., metakaolinite remains with a skeletal structure similar to that of the original mineral. At about 980°C., mullite nucleation begins and, as the temperature rises, more and more mullite crystals are formed, whilst a glassy phase forms from the fluxes and silica. Excess silica from the clay breakdown may form cristobalite at above 1,250°C., but little of the quartz in the original mixture is likely to be converted to this crystal form. Some of the quartz and of the amorphous silica from the clay decomposition may dissolve in the glassy phase and so influence its viscosity. Some advantage in vitrification may accrue from firing under slightly reducing conditions at the peak temperature. Cooling from the peak firing temperature should be slow to allow annealing of the glassy phase and particular care should be taken when passing through the β-α quartz inversion (at 573°C.) and the β-α cristobalite inversion in the range 230° to 170°C. The latter inversion involves a volume change roughly three times as great as that occurring in the β-α quartz inversion, and clearly the presence of too much cristobalite would be deleterious from the viewpoint of thermal shock of the finished ware. The aim in firing should therefore be to keep the cristobalite content to a minimum, involving careful apportioning of the ingredients and control of the firing conditions.

To increase the mechanical strength of the finished ware, an ideal would seem to involve the formation of a mat of small interlocking mullite needles whilst sufficient glass must be present to render the ware impermeable.

After cooling to ambient temperatures, the ware is carefully inspected and, where necessary, is machined to size to give pieces of the close dimensional limits necessary for building large-scale precision equipment.

Manufacture of porcelain

The basic composition is identical with the traditional Continental hard-paste porcelain formula 50% kaolin, 25% quartz, 25% felspar, but the methods of achieving the composition can differ considerably. For example, the renowned Berlin laboratory porcelain is compounded from Halle kaolin and Norwegian felspar, no quartz being deliberately added.[5] This kaolin contains appreciable amounts of free silica, some of which is in a very finely divided state, and the process seems to be to levigate the kaolin to remove all coarse quartz grains, leaving the very fine silica to supply the necessary compositional requirements. This seems to be in the range 22 to 25% of fine silica. The object of removing the coarse quartz is to give more complete vitrification during firing and to increase thermal shock resistance, coarse quartz grains being sources of mechanical weakness because of cracks formed during the inversions α-β quartz in the heating cycle and β-α quartz in the cooling cycle.

Another method of overcoming the coarse quartz problem is adopted at a second factory where the whole body is milled for twice the time normally adopted for tableware porcelain, whilst a third process involves elaborate screening devices to remove coarse quartz particles.

Making methods again include jolleying, with turning to give precision, and slip casting, but followed by careful drying before firing. As is normal in porcelain practice, the ware is given a light biscuit firing to about 900°C., after which it is dipped in a felspathic glaze and body and glaze are matured together in the glost firing which is around 1,400°C. A specially hard glaze is used in the case of the Berlin porcelain. An essential feature of porcelain firing is the reducing atmosphere which obtains around peak temperature; this allows complete vitrification at a somewhat lower temperature than would be necessary under oxidising conditions.

As with chemical stoneware, the ware can be polished and ground to size where necessary but, owing to the inferior plastic properties of the porcelain body, the size of articles that can be produced is more limited.

Acid-resistant brick and tiles

The composition varies according to the quality of the stoneware required. For coarse brickwork, red or blue engineering bricks are satisfactory. These are made from natur-

[Courtesy: Royal Doulton Potteries

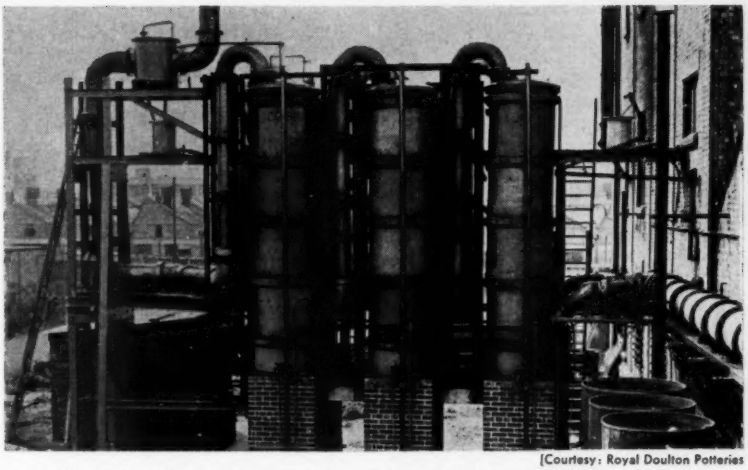
Fig. 6. Finishing a stoneware tower section in clay state, before firing

ally occurring surface clays, usually by plastic processes such as stiff-plastic pressing or extrusion from a pugmill and wire-cutting the issuing slugs.

For stiff-plastic blue bricks, the ware is set directly in the kiln from the press, and firing takes from 10 to 14 days, of which six to eight days are required for 'water smoking' to remove all moisture and achieve clay breakdown (500° to 550°C.), then follow 1½ to 2 days for the pre-heating to top temperature and, finally, 2½ to 3 days for full firing and the finishing fire under reducing conditions. Most blue bricks are now made by the wire-cut process and are dried on hot floors or in chamber dryers before setting in the kiln in the 'white-hard' state. Firing then takes six to eight days, of which 2½ to 4 days are occupied in water-smoking. Two methods of firing may be adopted from the water-smoking stage onwards. Traditionally the fires are built up rapidly and firing is completed with closed fires towards the end of the process in which no air space is allowed over the fuel bed. This procedure results in heavy smoke emission each time the fireholes are baited after the water-smoking. The other method involves so-called oxidising conditions of firing, an air space being left over the fuel bed, and these conditions are allowed to continue until the temperature at the top of the kiln attains 1,000° to 1,050°C. The fires are then closed and reducing conditions applied for the last 20 to 40 hours of firing.[6]

The metallic appearance of blue bricks was thought for a long time to be due to the development of ferrous silicates during the reducing firing, but recent work by Roberts, Riley and Rowden[7] has demonstrated that the appearance is due to reflection from a well-developed skin of haematite crystals. Red engineering bricks are fired without the special precautions for reducing conditions.

According to Vasel,[8] white-burning stoneware clays are especially suitable for the manufacture of acid-resistant bricks. Free silica must be absent, since it greatly impairs both mechanical strength and chemical resistance. Resistance to thermal shock is enhanced by the formation of mullite crystals and this appears to be favoured by the presence of suitable fluxes. Small crystals are formed in the presence of small amounts of fluxes whereas large amounts of flux favour the production of large crystals with consequent lower homogeneity and an impaired resistance to thermal shock,



[Courtesy: Royal Doulton Potteries

Fig. 7. Battery of chemical stoneware towers for washing fumes and reclaiming acid

a property that is also dependent on the size and shape of any grog incorporated in the ware. The free silica in the body can be removed by using clays containing lime or by using felspars or pegmatites as fluxes to dissolve the uncombined silica.

Porous ceramics

Porous filters are manufactured in various grades for use as filtering media. Kieselguhr, quartz, retort coke and vitrified grog may be used for preparing the granules, which are then carefully graded to the size limits required by the several grades, each with a uniform pore system. When the ware is to be fired to a high temperature, the ware may be pressed, moulded or slip cast, either with or without a binder such as sulphite lye and sufficient flux to form a bond at the firing temperature. Sometimes carbonaceous material such as wood flour may be added, which on firing burns out to give the required porosity.

Filter candles for the food, pharmaceutical and chemical industries have been made from a body consisting essentially of kieselguhr which is mixed with asbestos fibre and powder and a proportion of wood flour;[5] some grades also contain kaolin. The porosity is varied in the different grades by varying the proportion of wood flour. Water is added to make the body plastic and the material is then extruded through metal tubes in the form of long cylinders which are cut to length, dried and turned to size on a lathe. A stationary inverted drill is used to bore the central hole of the cylinder, which is rotated while held in a metal case. Firing is to 1,100° to 1,200°C. in intermittent kilns. The main drawback of such filters is their sensitivity to hydrofluoric acid, but high acid-resistance can be obtained by using graded alumina grog bonded with limited amounts of silica ester (ethyl silicate) of from 1.5 to 5-7 g./sq.m. of surface.[9] Such products are stated to be highly suitable for high-quality filters, the porosity of which can be altered by varying the grading of the alumina and the ethyl silicate content.

Inertness to attack

Perhaps the most striking feature of ceramics in relation to chemical plant is their inertness to attack. In a corrosion chart issued in 1959,[10] the relative reactivities of a whole range of metals, plastics and rubber, and other miscellaneous materials used in chemical plant were compared at three temperatures, 20°, 60° and 100°C. A wide variety of chemical agents were tested, both organic and inorganic, some 119 materials or types being considered. These included such vigorous reagents as the strong mineral acids and aqua regia, caustic alkalis and corrosive gases such as chlorine, fluorine and the sulphur oxides.

It is a striking tribute to chemical stoneware and chemical porcelain that attack at all three temperatures was only observed with fluorine and hydrofluoric acid, by alkalis in concentration of 10% or over at temperatures above the ambient, and by aliphatic halogen compounds such as chloroform at 100°C. Some attack at 100°C. was observed with 95% phosphoric acid; the remaining chemicals gave no evidence of reaction at any of the three temperatures listed. Comparable resistance was shown only by the expensive metals platinum (which reacts with aqua regia) and tantalum,

[Courtesy: Hathernware

Fig. 8. 'Armoured' chemical stoneware vessel

and by the plastics material PTFE. When these facts are taken in conjunction with the relative costliness of the different types of plant and, with the knowledge that chemical stoneware in particular can be fashioned in sizes as large as 6 ft. diam. and 8 ft. tall, it becomes self-evident that, for many chemical reactions, ceramics afford most valuable and relatively cheap materials for the manufacture of chemical plant. Regarding price, Hodson[11] stated that at that time (1950) whereas most corrosion-resistant materials cost many shillings/lb. weight, first-quality acid-resistant bricks cost only ½d./lb. and chemical stoneware 8d./lb. It is perhaps significant that a firm manufacturing highly corrosion-resistant alloys should use chemical stoneware in an acid-reclamation plant (Fig. 7).

New developments

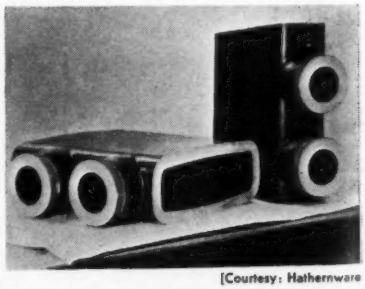
Although alumino-silicate materials are subject to attack by fluorine and hydrofluoric acid, ceramics are by no means confined to this field of materials and there is no reason why products should not be developed that show adequate resistance to fluorine derivatives. The development of porous ceramics based on alumina has already been indicated,[9] and a second development is of considerable significance in this respect. Reinhart[12] reports that a German patent has been filed that describes a method of producing tiles for chemical containers by sintering a material that is highly resistant both to hydrofluoric acid and to alkalis, thus countering two weaknesses of conventional chemical stoneware and porcelain. The composition involves fusing fluorides of Group II metals with glass-forming oxides of Group IV; the product of such an interaction may set to a clear glass. The hardened material can be crushed and ground and then shaped by normal making methods after the addition of a suitable plasticiser such as cellulose nitrate. The articles are vitrified at a temperature above the softening point and have the virtues of low firing shrinkage and considerable hardness.

Another weakness of conventional ceramic chemical plant is considered to be lack of mechanical strength. This can be countered in a number of ways: the conventional stoneware may be enclosed in a metal armouring sheath as illustrated in Fig. 8, metallic fillers such as ferro-silicon can be incorporated in the body which is then worked and fired in the conventional manner giving, it is claimed,[13] improved mechanical and refractory properties, or entirely new types of body, such as sintered alumina, have been developed with strengths equal to those of regular cast irons and which increase the life of equipment such as rotary seals, reciprocating pumps, control valves and metering or mixing nozzles.[14]

Perhaps the gravest objection to ceramics is their sensitivity to temperature differences, particularly if these are suddenly imposed. Conventional stoneware will probably withstand temperature differences between faces of the order of 30° to 40°C., but newer developments will allow differences of as much as 100°C.[11] Improved resistance to thermal shock can be obtained either by reducing the thermal expansion of the body or by improving the thermal conductivity. Thermal expansion can be reduced by incorporating talc in the body, which then develops a proportion of cordierite of exceptionally low expansion characteristics. Cordierite bodies can be prepared with a thermal expansion less than half and a tensile strength twice that of the conventional ware.[15] This tendency can now conceivably be carried further; the use of lithium compounds in the composition results in the formation of lithium aluminium silicates with thermal expansions that can be varied over a very considerable range. Indeed it is possible to produce materials with virtually zero thermal expansion over a considerable temperature range.

The thermal conductivity of ordinary chemical stoneware between 0° and 500°F. is about 1 Kcal/m.hr.°C. and this cannot be increased to compete with metals, where high conductivity is an advantage, except by the incorporation of a 'metallic grog'. Thermal shock resistance may be increased by adding fused alumina, silicon carbide or mullite, which at the same time improve the mechanical properties.[16] Kingsbury[15] reports that, by replacing a large proportion of the siliceous materials by silicon carbide, ware can be produced with a thermal conductivity about 300% greater than that of conventional stoneware. The way in which the properties of stoneware have been improved is illustrated by Table 2[16] in which Robitschek compares the properties of ordinary stoneware with those of the best values obtainable in special stoneware bodies.

For some special problems in the chemical industry the development of the so-called super-refractories has offered a new solution. Essentially super-refractories embrace ceramic and self-bonded kiln-fired products, made from silicon carbide, fused alumina and electric furnace mullite as well as electrically-fused cast-refractory materials.[17] Table 3 lists some physical properties of certain of these materials, a striking feature being the high temperatures which these materials can withstand. The materials offer high thermal conductivity (particularly with silicon carbide materials) or resistance to thermal shock, corrosion resistance,



[Courtesy: Hathernware

Fig. 9. Porcelain segments of synchrotron 'doughnut'

The material of construction for chemical plant which will be discussed in next month's issue of CHEMICAL & PROCESS ENGINEERING will be

NEW METALS

Table 2. Comparison of conventional and new stoneware bodies[16]

	Ordinary stoneware	*Maximum values in special stonewares*
Compressive strength, p.s.i.	59,700	129,400
Tensile strength, p.s.i.	1,350	8,820
Modulus of rupture, p.s.i.	5,120	17,920
Impact strength, cm. kg./c.c.	1.7	4.7
Abrasion loss, c.c.	6.6	1.6
Thermal expansion, /°C.	2.6×10^{-6}	0.15×10^{-6}
Thermal conductivity, K cal./m.hr.°C. ..	0.98	5.2

Table 3. Physical properties of bonded and fused cast refractories[17]

	Silicon carbide	*Silicon carbide (silicon nitride bonded)*	*Fused alumina (low silica)*	*Mullite electric furnace*	*Fusion-cast α/β-alumina*
Max. safe working temp., °C.	1,750	1,800	1,800	1,800	1,875
Porosity, %	13.2	7.9	21.9	22.7	2.5
Modulus of rupture, 1,350°C.	200	5,640	200	433	n.d.
Abrasion resistance	Excellent	Excellent	Fair	Fair	Good
Relative resistance to spalling (thermal shock resistance)	High	Very high	Good	Very good	Low

high strength and resistance to abrasion or erosion.

Particular applications of silicon carbide are cooling and scrubbing towers, spray nozzles, venturi mixers or feeders, agitator and mixer blades, pumps, diffusers and cyclone collector linings. For example, silicon carbide will withstand immersion in hot corrosive melts such as aluminium for die casting which, although really a metallurgical operation, is indicative of the qualities of the material. Electrical furnace mullite has been used for structural piers where a strong, inert and heat-shock resistant, high-temperature material is necessary.

Special advantages

To illustrate the adaptability of ceramics to chemical and allied problems we have only to remember that the apparatus for growing *penicillium notatum* in penicillin production and the apparatus for filtration was stepped up from laboratory-scale ware to full-scale production, and that articles as diverse as chromatographic tanks and the sections for the accelerator of the synchrotron at Birmingham University are made of ceramic materials. This last example is worthy of further comment because it illustrates the precision with which ceramics can be produced, although the problem on this occasion was one of nuclear rather than chemical engineering.[18] The synchrotron was designed to produce atomic particles acquiring an energy of 10^9 electron-volts whilst circulating in a vacuum chamber of diameter 450 cm. and cross-section 40 × 10 cm. The ' doughnut ' was required to have the greatest electrical resistance possible, to have a smooth glaze and certain sections were to be given a silver and others a platinum coating. One length of the ' doughnut ' was to be larger in section than the rest, to accommodate intake and offtake sections. The problem was solved by dividing the doughnut into a 30-sided polygon each side of which constituted two sections as illustrated in Fig. 9. The work was carried out by nuclear engineers in collaboration with two producers of ceramics.

The sections were slip-cast from a hard porcelain body with wall thicknesses of 3 cm. and a non-conducting glaze was applied to the dry sections, which were then fired in a gas-fired tunnel kiln to 1,250°C. The sections were then machined by silicon carbide wheels to tolerances of ±0.1° on the angles and ±0.1 mm. in length and were then hand-lapped. Each section was tested under vacuum and the silver or platinum applied to the inner surfaces before enamel firing.

Ceramics are advancing into new fields, particularly where exceptional temperatures and abrasion conditions are being encountered as, for example, in the nose cones (radomes) of missiles and the leading edges of supersonic aircraft. These developments are giving ever greater knowledge of the properties of ceramic materials and how they may be modified, knowledge that will redound to the benefit of the more conventional ceramic uses, of which adaptation to chemical plant is a most important example.

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Polyurethane foam

Houdry Process Corp. of Philadelphia and Compagnia Internazionale Generale Industriale-Malatesta SpA. have formed an equally owned company to manufacture and sell polyurethane foam and foam products.

The new firm, Montecassino SpA., is located in Cassino, Italy. Manufacturing operations in the southern Italy city are expected to start later this year. The establishment of the Italian firm is the second European joint venture effected by Houdry in the past 10 months. In September, the Philadelphia company and Chemische Werke Huels of Marl, West Germany, formed Katalysatorenwerke Houdry-Huels GmbH. to manufacture and sell catalysts.

Initially, Montecassino will market its foam and foam products in southern Italy and the Mediterranean basin area. Both rigid and non-rigid polyurethane foam will be manufactured, and the product line will include a wide range of foam items.

Houdry has been engaged in the urethane foam market for the past three years through the manufacture and sale of the catalyst triethylenediamine.

In addition to Montecassino and K.W.H., Houdry has several wholly owned, partially owned and affiliate companies.

Evaporation Plant Design—Furfural and Drying Oil

By F. Molyneux,* Ph.D., B.Sc.(Lond.), B.Sc.(Eng.), A.M.I.Chem.E., A.M.I.Mech.E.

The importance of furfural as a selective solvent in mineral oil refining has been recognised for some time by the paint industry. In order to recover furfural and separate the drying oil it is usual to subject the extract to evaporation in a climbing-film evaporator. This article describes the design of such an evaporation plant to handle 4,470 lb./hr. feed. In order to allow for minimum vapour velocity of 30 ft./sec. the evaporator tube length was found to be 15 ft. Other considerations discussed include steam distillation, entrainment and furfural recovery.

THE use of furfural as a selective solvent in mineral oil refining has been established for some time, and its use as a selective solvent to obtain from soya-bean oil a fraction containing the unsaturated constituents for use as a drying oil is becoming of increasing importance, the drying oil produced threatening the position previously held by linseed oil in this field.[7, 8]

Furfural, or furfural naphtha, has been found to be the most selective solvent, and experiments have shown that a single batch extraction can produce an extract of 145 iodine number from soya-bean oil of 130 iodine value.

To recover the furfural and to separate the drying oil the extract is subjected to evaporation in a climbing-film evaporator, since if solvent recovery were attempted by the usual methods of distillation it is found that the oil contains anti-oxidants due to traces of furfural and to products resulting from the decomposition of furfural. The design of a suitable climbing-film evaporator for this duty is considered in the following article.

Climbing-film evaporator

This type of evaporator, originally patented by Kestner in France, has come into increasing use where an evaporation is required to be once-through with a high rate of heat transfer and consequently high velocities.

The tubes may be $\frac{3}{4}$ to 2 in. diam. and from 10 to 25 ft. long, the ratio of length to diameter being over 125. The boiling is used to obtain high liquid velocities by dragging a film of liquid up the walls of the tube with the rising vapour. The mixture of unevaporated liquid and vapour emerges from the tubes at high velocity and strikes a baffle in a separating chamber, where disengagement takes place.

The unit therefore is particularly suitable for foaming liquids and the low residence time in the heating tube (10 to 20 sec.) reduces the danger of decomposition of heat-sensitive materials. The high velocity in the tubes also inhibits scale formation and prevents deposition of mucillageous materials, providing that the design ensures that the top of the tubes are not left dry due to complete vaporisation. McNelly[4] suggested that a maximum figure of 80% vaporisation was sufficient to ensure that this would not occur.

The present tendency is to use once-through operation of the evaporator without recirculation and to feed the liquid to the evaporator at its boiling point, thereby greatly increasing the capacity, since practically the whole length of the tube may be used for evaporation.

The unit is particularly suitable for vacuum operation, as the overall height is 30 to 35 ft. and can be conveniently operated with a high-level jet condenser and a barometric leg. A detachable cover over the disengagement section, or immediately above the calandria, is usually fitted to enable tube cleaning.

The main difference between evaporation and distillation lies in the fact that in evaporation the prime interest is in the removal of one constituent from the solution as a vapour and, in the present case, the evaporation desired is such as to completely strip the solvent furfural from the oil and, to do this, steam stripping of the oil must proceed continuously to remove all the solvent.

Consequently, this operation is performed with superheated steam in the entry to the disengagement space, and the resulting furfural steam mixture condensed out in a tubular condenser and finally separated in a solvent water separator.

Design problem

It is required to separate a furfural drying oil mixture obtained as an extract from the separation of a drying oil of high iodine value from a solvent-extracted soya-bean oil. The semi-technical plant feed consisted of 4,470 lb./hr. containing 470 lb. drying oil (MW = 283) and 4,000 lb./hr. furfural (MW = 96). To keep the furfural within the accepted evaporation figure for climbing-film evaporators, it is only 95% evaporated in the tubes, the remaining furfural being stripped from the oil by a venturi scrubber fed with superheated low-pressure steam.

The feed passes through a Brown fin-tube preheater

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before entering the evaporator, where it is heated to its boiling point at the inlet pressure conditions. A pressure of 50 mm. Hg is maintained in the disengagement space and the evaporation is carried out by means of steam condensing in a shell and tube heat exchanger. Assuming a steam heat-transfer coefficient of 2,000 B.T.U./hr.sq.ft.°F., what surface area will be required for the evaporation and what will be the steam distillation requirements?

Properties of furfural

Density: At 20°C. = 1.1598
At 25°C. = 1.1545

Heat of vaporisation $\Delta H_{433.8}$ *K*cal./mol. = 10.22

Specific heat liquid, cal./g.°C.: 14— 80°C. = 0.401
20—100°C. = 0.416

Thermal conductivity, B.T.U./sq.ft.hr.°F./ft. at 100°F. = 1.525

Viscosity, centipoises: At 0°C. = 2.48
At 25°C. = 1.49
At 38°C. = 1.35
At 54°C. = 1.09
At 99°C. = 0.68

Surface tension, dynes/cm.: At 0°C. = 43.5
At 29.9°C. = 40.7
At 30.0°C. = 41.1

Vapour diffusion coefficient, sq.cm./sec.:
At 17°C. = 0.076
At 25°C. = 0.087
At 50°C. = 0.017

Properties of drying oil

Density = 0.8836 + 0.0002879*T*°F. g./c.c.
Specific heat = 0.45 + 0.000626 (*T*°F. – 60) B.T.U./lb. °F.
Thermal conductivity = 0.0808 B.T.U./sq.ft.hr.°F./ft.
Viscosity = 11.2 cp. at 100°F., 2.5 cp. at 210°F.
Tube diameter = 1 in. o.d. stainless steel

Pepper[2] gives minimum feed rate $= \dfrac{4{,}470 \times 2}{3}$

$= 3{,}000$ lb./hr.

Minimum vapour $= \dfrac{3{,}000}{3}$ $= 1{,}000$ lb./hr.

$= \dfrac{1{,}000 \times 359}{96} \times \dfrac{391}{273} \times \dfrac{1}{3{,}600}$ $= 1.5$ cu.ft./sec.

Surface area at minimum vapour velocity, 30 ft./sec.

$= \dfrac{1.5}{30}$ $= 0.05$ sq.ft.

Expressing the flow rate as a mass velocity, then

$$G = \frac{4{,}470}{0.05} = 89{,}400 \text{ lb./sq.ft.hr.}$$

The heat-transfer equation is:

$$\int_v^{\Delta} dA = \int_o^q \frac{dq}{U \Delta t}$$

dq may be evaluated by the vaporisation of the furfural

$$dq = dW_f \lambda$$

where W_f is feed rate of furfural in lb./hr. and λ is the latent heat of evaporation in B.T.U./lb.

Then $$A = \int_0^{W_f} \frac{\lambda dW_f}{U_i \Delta t}$$

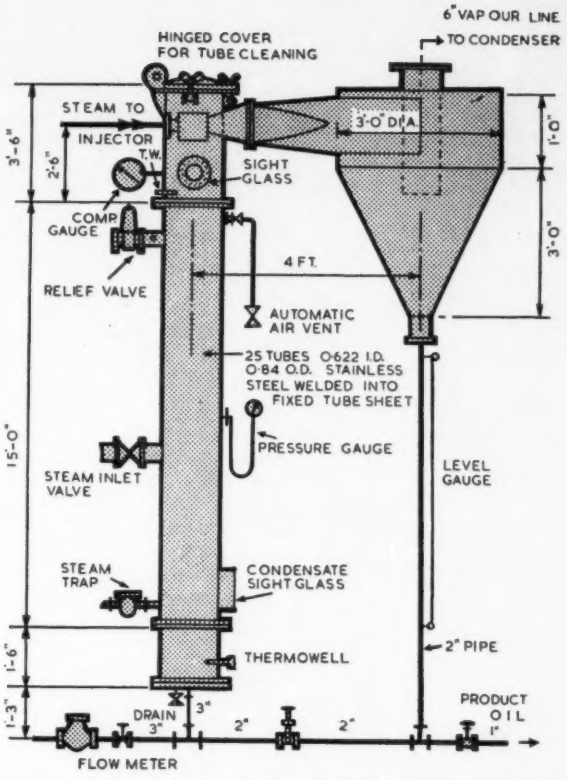

Fig. 1. General arrangement of climbing-film evaporator, steam ejector and cyclone separator, made in stainless steel

In many simple cases of evaporation the r.h.s. of this equation could be integrated analytically assuming constant values of λ and U_i. However, in this case, both λ and U_i vary with temperature to an appreciable extent and it is necessary to evaluate them at several temperatures between those of inlet and outlet. The equation is then integrated graphically using point values of $\lambda / U_i \Delta t$.

The overall resistance based on the inside surface area is:

$$\frac{1}{U_i A_i} = \frac{1}{h_i A_i} + \frac{\Delta x}{K A_m} + \frac{1}{h_o A_o}$$

$$\frac{1}{U_i} = \frac{1}{h_i} + \frac{\Delta x \; A_i}{K A_m} + \frac{A_i}{h_o A_o}$$

For circular cross-sections A is proportional to D, then:

$$\frac{1}{U_i} = \frac{1}{h_i} + \frac{\Delta x \; D_i}{K D_m} + \frac{D_i}{h_o D_o}$$

$D_i = 0.622$ in.
$D_o = 0.840$ in.
$D_m = 0.731$ in.
$\Delta x = 0.109$
$K_{stainless} = 11$ B.T.U./hr.sq.ft.°F./ft.

For thin-walled tubes the arithmetic mean is satisfactory, so that

$$\frac{1}{U_i} = \frac{1}{h_i} + \frac{(0.109/12)\ 0.622}{(11)\ (0.731)} + \frac{0.622}{(2{,}000)(0.840)}$$

$$\frac{1}{U_i} = \frac{1}{h_i} + 0.00107$$

This is a general equation expressing the overall resistance in terms of the inside resistance.

At any point within the exchanger $\Delta t = (170 - T°C.) = 1.8\,(170 - T°C.)°F.$, where T is the bulk average temperature of the drying oil and furfural mixture at that point. Points in the evaporator corresponding to temperatures between inlet 95°C. and outlet 145°C. will be selected at 110°C., 120°C., 130°C. and 140°C.

Heat-transfer correlations

The Coulson and McNelly correlations for low heat flux were given:[4]

$$\mathrm{Nu} = (1.3 + 39d)\,\mathrm{Pr}^{0.9}\,\mathrm{Re}_L^{0.23}\,\mathrm{Re}_V^{0.34}\left(\frac{\rho_L}{\rho_V}\right)^{0.25}\left(\frac{\mu_V}{\mu_L}\right)$$

Nu = Nusselt group
Pr = Prandtl group
Re = Reynold's group
d = tube diameter, ft.
ρ = density, lb./cu.ft.
μ = viscosity, cp.

L and V denote liquid and vapour phase respectively.

Points in the evaporator corresponding to bulk temperatures of 95°C., 110°C., 120°C., 130°C., 140°C. and 145°C. are selected (see Table 1).

The inside coefficient of the mixture is determined using the above correlation.

In the present case the liquid is preheated to boiling point at the pressure appertaining at the entrance to the evaporator tube. The proportions of vapour and liquid will vary along the tube, being dependent on both pressure and temperature at the point in the tube.

The pressure-drop profile along the tube can be determined by the correlation of Lockhart and Martinelli,[11] but a trial and error method must be used working back from the outlet conditions at the top of the evaporator tube.

At 95°C.

Evaporator tube inlet pressure = 150 mm. Hg. $(\mathrm{Re})_V$ is negligible since vapour formation is only just commencing.

$$(\mathrm{Re})_L = \frac{dG}{\mu_L} = \frac{0.622}{12} \times \frac{89{,}400}{0.68 \times 2.42} = 2{,}830$$

$$\mathrm{Pr} = \frac{c\mu}{k} = \frac{0.416 \times 0.68 \times 2.42}{0.1525} = 4.5$$

$$\text{Then} \quad h_i = \left[1.3 + 39\left(\frac{0.622}{12}\right)\right] \frac{0.1525 \times 12}{0.622} \times (\mathrm{Pr})^{0.9}(\mathrm{Re}_L)^{0.23}\left(\frac{\rho_L}{\rho_V}\right)^{0.25}\left(\frac{\mu_V}{\mu_L}\right)$$

$$\text{where} \left(\frac{\rho_L}{\rho_V}\right) = \frac{1.545 \times 62.4 \times 359 \times 368}{96 \times 273} = 367$$

$$\text{and} \quad \left(\frac{\mu_V}{\mu_L}\right) = \left(\frac{0.0135}{0.68}\right) = 0.0267$$

$$h_i = \frac{0.1525 \times 12}{0.622} \times \frac{3.33 \times 3.89 \times 6.281 \times 4.3 \times 0.0267}{}$$

$$= 31.7 \text{ B.T.U./sq.ft.hr.°F.}$$

$$\frac{1}{U_i} = \frac{1}{h_i} + 0.00107$$

$$\frac{1}{U_i} = 0.0315 + 0.00107 = 0.03257$$

$$U_i = 31.3 \text{ B.T.U./sq.ft.hr.°F.}$$

At 145°C.

$$(\mathrm{Re})_L = \frac{0.622}{12} \times \frac{9{,}900}{0.68 \times 2.42} = 314$$

$$(\mathrm{Re})_V = \frac{0.622 \times 80{,}000}{12 \times 0.0195 \times 2.42} = 88{,}000$$

$$h_i = \frac{0.1525}{0.622} \times 12 \times 3.33 \times 3.89 \times 3.274 \times 47.86 \times 4.5 \times 0.0287$$

$$= 648 \text{ B.T.U./sq.ft.hr.°F.}$$

$$\frac{1}{U_i} = 0.00152 + 0.00107 = 0.00259$$

$$U_i = 385 \text{ B.T.U./sq.ft.hr.°F.}$$

This same sequence is repeated for each of the other temperatures.

Using $$\lambda_2^1 = \lambda_1\left[\frac{1 - (T_2/T_c)}{1 - (T_1/T_c)}\right]^{0.38}$$

the critical temperature for furfural is 397°C. Then

$$\lambda_2^1 = 192\left(\frac{397 - 145}{397 - 95}\right)^{0.38}$$

$$= 192 \times (0.835)^{0.28} = 178$$

To determine the area required involves integration of the equation

$$A = \int_0^W \frac{\lambda dW}{U_i(-\Delta t)}$$

The integration is performed graphically by plotting $\lambda/U_i(-\Delta t)$ against values of W and determining the area under the curve between the limits $W = 0$ and $W = 3{,}975$ lb., and a value of 56.8 sq. ft. is obtained.

The tube bundle specified is: shell diameter, 8 in.; number of tubes, 25; tube inside diameter, 0.622 in.; tube outside diameter, 0.840 in.; pitch, $1\frac{5}{16}$ in. triangular. The length of the tube between tube sheets is therefore:

$$\frac{56.8}{25 \times \pi} \times \frac{12}{0.622} = 14 \text{ ft.}$$

To allow for scaling up with mucillageous material, the length of the tube is fixed at 15 ft. In operation overall coefficients were obtained, varying from 115 to 125 B.T.U./sq.ft.hr.°F., and these are in general agreement with those reported in the literature by Gloyer[8] and Cessna, Lientz and Badger[13] for similar duties. The pressure drop/ft. is 1 to 2 mm. for the first 10 ft. of the tube, with the remaining 80 mm. pressure drop taking place in the last 5 ft.

Table I.

T°C.	$-\Delta t = (170 - T)1.8$	h_i	U_i	λ B.T.U./lb.	lb.	$\frac{\lambda}{U_i(-\Delta t)}$
95	135	31.7	31.3	192	0	0.0455
110	108	154.6	130	187.8	275	0.0134
120	90	182	152	185	700	0.01338
130	72	317	236	182.2	1,000	0.0107
140	54	505	326	179.4	1,000	0.0102
145	45	648	386	178	1,000	0.0102

Total = 3,975 lb.

Effect of concentration

When a solution is evaporated, not only are the effects of liquid depth and acceleration present, but the effect of concentration on the boiling point must be considered. For ideal solutions the concentration effect may be estimated through Raoult's and Dalton's laws, for $P = p_a + p_b = P_a X_a + P_b X_B$, where a and b refer to solute and solvent respectively. If the solute is non-volatile then, as in the present case, $P = P_b X_b$.

Defining P° as the total pressure of the vapour over liquid phase, then for the pure solvent $P^\circ = P_b$, since $X_b = 1$, and the vapour pressure lowering due to the pressure of solute becomes

$$\frac{P^\circ - P}{p^\circ} = \frac{1 - X_b}{1} = X_a$$

$$T_B - T_B{}^\circ = KX_a$$

where T_B = boiling point of solution and $T_B{}^\circ$ = boiling point of pure solvent.

The pressure profile in the evaporator tube is determined by the correlation of R. W. Lockhart and R. C. Martinelli[11] applied to isothermal two-phase two-component flow in pipes, but the concentrations and temperature profile can be determined by trial and error.

The method of determining the latent heat of solution which shows a considerable rise in boiling point can be determined as follows.

If in the Clapeyron equation it is assumed that the volume of liquid is negligible compared with that of the vapour and the vapours conform to the ideal gas laws, then

$$\frac{dp}{dT} = \frac{L}{T\Delta V} = \frac{pL}{RT^2}$$

Similarly, for a reference liquid usually water at the same vapour pressure

$$\frac{dp}{dT_R} = \frac{pL_R}{RT_R{}^2}$$

Then $$dp = \frac{pL\ dT}{RT^2} = \frac{pL_R\ dT_R}{RT_R{}^2}$$

Then $$L = \lambda = L_R \frac{T^2}{T_R{}^2} \frac{dT_R}{dT}$$

The value of dT_R/dT is obtained as the slope of the Duhring line of vapour pressures at the same temperature. This procedure is repeated for each temperature and concentration. An alternative expression is

$$\lambda_2 = \lambda_1 \left[\frac{1-(T_1/T_c)}{1-(T_2/T_c)}\right]^{0.38}$$

where T_c = critical temperature.

Tube diameter and length

The diameter and length of tube in climbing-film evaporators has been investigated by Pepper,[2] who reports that 100 ft./sec. is a suitable working velocity for evaporations at atmospheric pressure. Another factor affecting the selection of the tube diameter is the minimum feed rate necessary for steady operation. Pepper found that for tubes up to 1 in. diameter this minimum feed rate is approximately three times the amount of liquid that needs to be evaporated to give the required minimum vapour

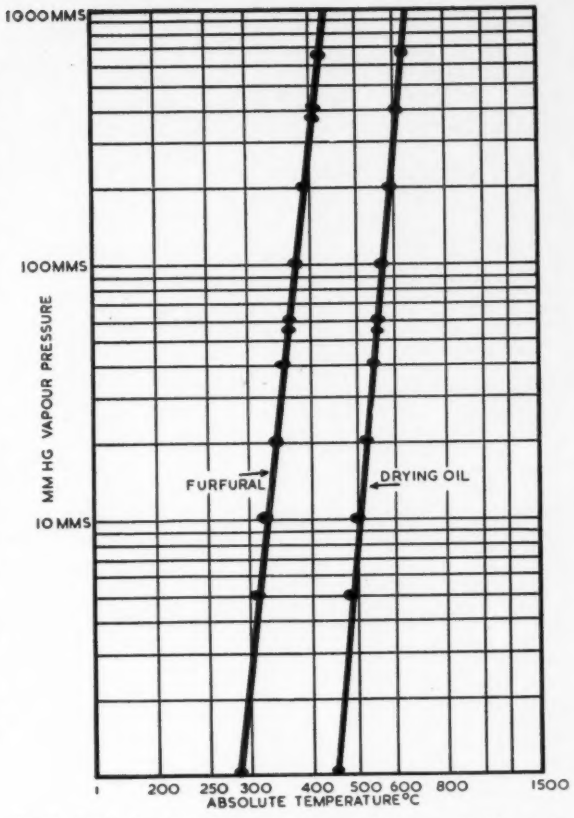

Fig. 2. Graph showing relationship between absolute temperature and vapour pressure, for furfural and drying oil

velocity of 30 ft./sec. in the tube. The recommended feed rate is 1.5 times the minimum.

The length of the tube must be such as to provide the required rate of heat transfer without too high a temperature difference. Drew and Mueller[12] recommended a temperature difference less than 25°C. across the boiling film and Hands and Norman[3] stipulated that a length 100 times the diameter usually gave satisfactory results, although the original Kestner specification was 125 diameters in length.

Evaporation factor

Dry patches on the top of the tube wall must be avoided by restricting the evaporation factor, and McNelly[4] suggested that to ensure this an evaporation of not greater than 80% should be attempted and that it was advisable to design for an evaporation factor not greater than seven.

Bearing all these factors in mind, the design of the evaporator tube bundle considered for this duty consisted of a stainless-steel tube welded into fixed stainless-steel tube sheets.

Details of the layout and disengagement, etc., are as shown in Fig. 1.

Steam distillation

The factors affecting steam requirements in steam distillation are determined from examination of the vapour pressure relationships.

Let N_s be the number of mols of steam in the vapour

of molecular weight M_s and weighing W_s required to distil $(N_b)_1 - (N_b)_2$ mols of volatile material of molecular weight M_b and weighing W_b from N_a mols of non-volatile material constant in the distillation.

N_b = number of mols of volatile material in the distillation at any one time.

Let p_s be the actual partial vapour pressure of steam in the vapour

p_b = actual partial vapour pressure of the volatile component in the vapour

p^1_b = equilibrium vapour pressure of the volatile component

P_b = vapour pressure of pure component at constant temperature.

By Raoult's law

$$p^1_b = P_b \frac{N_b}{N_a + N_b}$$

and vaporisation efficiency

$$E = \frac{p_b}{(P_b N_b)/(N_a + N_b)}$$

Let P be the total pressure and temperature T be constant so that $P = p_s + p_b$.

Assuming no water is present in the distillation, so that p_s is less than the saturation pressure of the steam at the temperature of the still, then, since volatile components distil at such ratios that their molecular proportions correspond to their vapour pressures for differential amounts

$$\frac{dN_s}{dN_b} = \frac{p_s}{p_b} = \frac{P - p_b}{p_b} = \frac{P}{p_b} - 1$$

Introducing the efficiency term E

$$p_b = E\, P_b \left(\frac{N_b}{N_a + N_b}\right)$$

$$dN_s = -\left(\frac{P}{EP_b} - 1\right) dN_b - \frac{PN_a dN_b}{EP_b N_b}$$

On integration

$$N_s = \left(\frac{P}{EP_b} - 1\right)[(N_b)_1 - (N_b)_2] + \frac{PN_a}{EP_b} \log e \left[\frac{(N_b)_1}{(N_b)_2}\right] \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

When there is a relatively small amount of non-volatile component the equation is simplified so that p_b can be considered constant

$$p_b = E\, P_b$$

$$\frac{dN_s}{dN_b} = \frac{p_s}{p_b}$$

$$N_s = \frac{p_s}{p_b}[(N_b)_1 - (N_b)_2]$$

$$\frac{N_s}{[(N_b)_1 - (N_b)_2]} = \frac{p_s}{p_b} \quad \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

In terms of weights

$$\frac{W_s}{W_b} = \frac{p_s}{p_b}\frac{M_s}{M_b} = \frac{(P - p_b)M_s}{p_b - M_b} = \frac{(P - EP_b)M_s}{E\, P_b\, M_b}$$

which is the usual form.

The amount of steam required for stripping is found by substitution of the correct quantities in equation (1). Steam used for the stripping is at 25 p.s.i.g. and is superheated to 280°F., expanding *via* a convergent/divergent nozzle to 100 mm. Hg pressure absolute.

Steam required

The amount of furfural to be stripped out is 25 lb. of molecular weight 96 from 470 lb. of oil of molecular weight 283. The equilibrium vapour pressure of the furfural is 370 mm. and the actual partial pressure is 50 mm. Hg.

An approximate value is given by equation (2). The weight of steam

$$W_s = \frac{100}{50} \times \frac{18}{96} \times 25 = 9\tfrac{1}{2} \text{ lb./hr. of steam.}$$

Steam for the evaporator section is supplied at 100 p.s.i.g. and the injection steam is bled off this line through a superheater and reducing valve.

Heat and mass balance

Using as a datum temperature that of the cooling water entering the condenser and time hours:

W_s = weight of steam used

L_s = latent heat of condensation of steam at temperature of condensation T_s

w_s = weight of steam condensate/hr.

W_1 = weight of feed liquor/hr.

W_2 = weight of concentrated liquor

w = weight of vapour

λ = latent heat of evaporation

T = datum temperature

T_1 = entering temperature of the feed liquor

T_2 = leaving temperature of concentration liquor (and of vapours)

Then $W_s[L_s + (T_s - T)] + W_1(T_1 - T)$
$= W_2(T_2 - T) + w[\lambda + (T_2 - T)] + w_s(T_s - T)$
$+$ heat loss from evaporator surfaces.

Including the condenser, the heat terms for the vapour may be replaced as follows:

W_3 = weight of cooling water

T_3 = leaving temperature of cooling water

Then

$$W_3(T_3 - T) + w(T_2 - T) = w[\lambda + (T_2 - T)]$$

If it is assumed that the steam condensate leaves at the steam temperature and the entering steam is not superheated, then the sensible heat term for the entering steam is cancelled by that of the condensate heat term. Similarly, a material balance is given by:

$$W_s + W_1 = w + w_s + W_2$$

Scale in evaporation

The overall heat-transfer coefficient must decrease as the scale builds up since an additional heat-transfer resistance is being formed in series with those already present.

$$U = \frac{1}{R_o + R_s} = \frac{1}{(1/U_o) + aQ}$$

U = overall coefficient at time θ after start of evaporation

U_o = overall coefficient at start of evaporation

R_o = resistance to heat transfer existing at start of evaporation

R_s = resistance to heat transfer resulting from scale formation

Q = total amount of heat transferred from time 0 to θ

a, b = constants

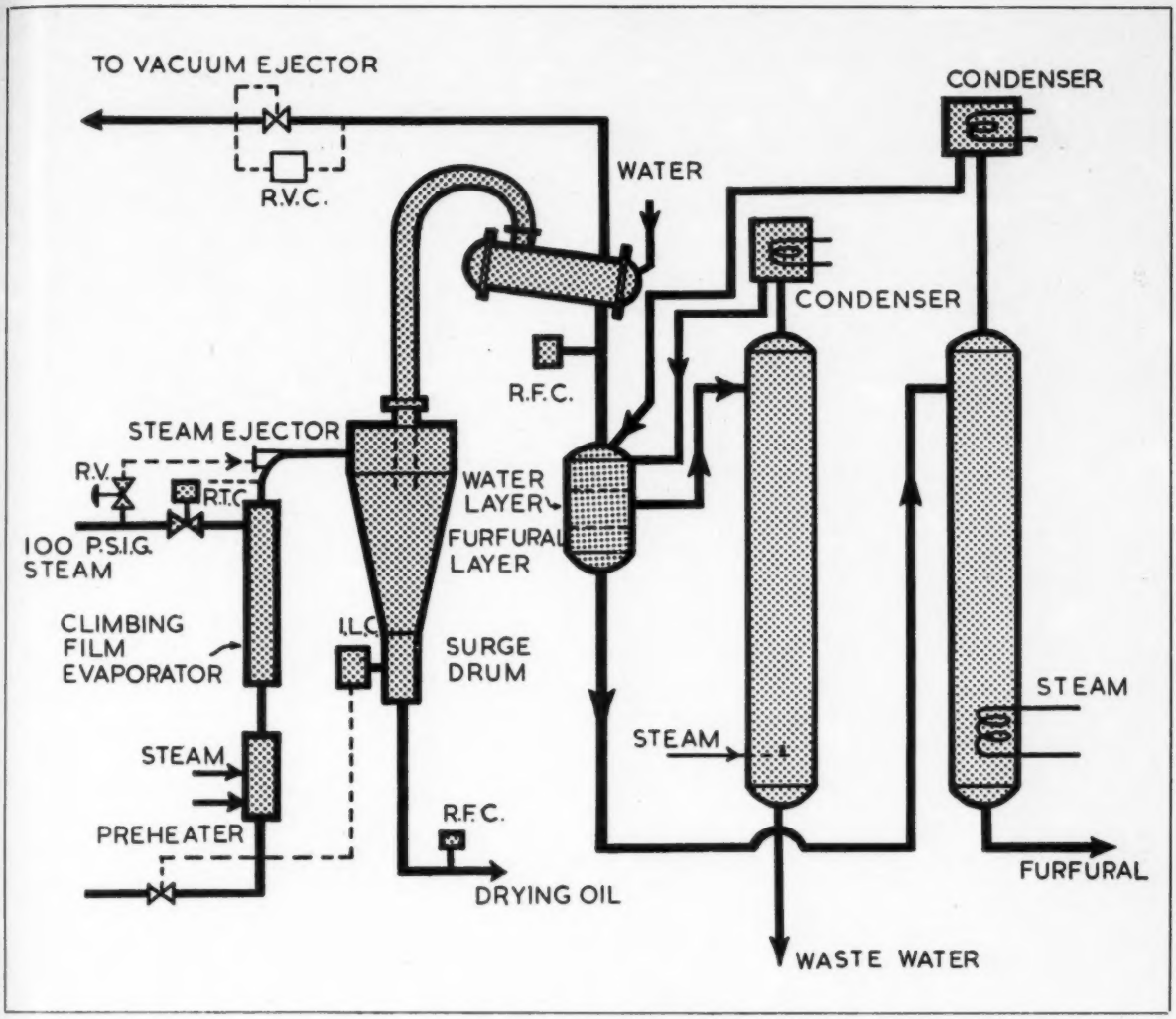

Fig. 3. Flowsheet of flash evaporator, steam ejector, cyclone separator and distillation columns

Overall coefficient

Then the overall coefficient at time θ can be proved to be given by an equation of the form:

$$\frac{1}{U^2} = a + b\theta$$

Thus, in practice, if this coefficient is determined at regular intervals the results can be plotted to determine the values of the constants a and b, and from these an accurate prediction can be made as to when the coefficient is going to exceed the designed value and thus necessitate maintenance cleaning.

Smith and Montross[9, 10] have given suitable methods of removing liquid droplets from a vapour. These can be divided into the following classes:

(1) Gravity settlers

In these the velocity of the gas stream is reduced in order to allow the droplets to settle on some surface. The lower limit of usefulness is droplets of 50 microns diameter.

(2) Inertial separators

The vapour stream is made to follow a bending path and the droplets are thrown on to the surfaces of the separator. The cyclone belongs to this class.

(3) Packed columns and filters

The vapour stream is passed through a column filled with suitable packing, the droplets being collected by diffusional and inertial processes.

In the present case, the most suitable equipment is provided by a cyclone with tangential inlet for steam furfural vapour and oil droplets.

Separation of 10-micron droplets is achieved in a stainless-steel cyclone 3 ft. diameter at the top of the cone with a 1-ft.-long cylindrical portion and a 3-ft.-long cone. The falling velocity observed for a 10-micron droplet in air is 0.008 ft./sec. The linear velocity of the entering vapour is 100 ft./sec. and a centrifugal effect of:

$$\frac{100^2}{1.5 \times 32.2} = 207 \text{ is attained.}$$

The velocity of the 10-micron droplet then becomes 1.66 ft./sec. outwards, and in 1 sec. it will have contacted the side of the separator and be then carried to discharge.

This time of 1 sec. gives the residence time regained for the vapour in the cyclone, and from this the cyclone volume. The proportions making up this volume can then be determined from those experimentally determined for maximum efficiency and can be shown to approximate those chosen.

The pressure drop across this cyclone will be the equivalent of some 5 to 6 in. w.g.

Feed preheater

It is essential for efficient operation that the feed enters the evaporator at its boiling point and, in the present case, this is ensured by the feed entering *via* a Brown fin-tube preheater, the design of which follows closely the methods demonstrated in a previous article.[5]

The solvent vapours and the superheated steam vapour are separated from the oil by means of a tangential-type vapour liquid separator, the vapours then passing to a horizontal drip tube type condenser with water inside the tubes, this condenser being designed in accordance with the treatment given by the author elsewhere.[6]

The liquor from the condenser is then fed to a decanter where it separates into two layers:

(1) A water layer containing approximately 8.8% furfural; and

(2) A furfural layer containing about 94.2% furfural.

Furfural recovery

The water layer is steam-stripped of its furfural in a small 15-plate column, giving an azeotrope vapour containing 20 to 25% furfural, which is returned *via* a condenser to the decanter. The furfural layer leaving the decanter will contain about 5.8% water. This solution may be dehydrated by continuous distillation using a second column. The overhead vapour is a mixture of furfural and water, whilst the liquid from the bottom of the column is dry furfural containing less than 0.05% water. The condensate is sent to the stripping column decanter, from which the water layer is returned to the top of the stripping column and the furfural layer to the top of the dehydrating column. Dehydration is usually carried out under vacuum in order to reduce losses due to polymerisation of furfural.

Materials of construction

Although normally mild-steel equipment is satisfactory for handling dry furfural and drying oil solutions, Hands and Norman[3] have reported serious corrosion, and particularly erosion, at the top of climbing-film evaporator tubes.

Since it is important to avoid discoloration and contamination in this case, it was decided to make the tube bundle and tube plates in stainless steel. The equipment following the steam ejector for steam stripping the oil of furfural is dealing with wet furfural and all surfaces in contact with this vapour must therefore be of stainless steel.

Comments on design

Kern in his 'Process Heat Transfer' places several limitations on the design of industrial evaporators and reboilers since he doubts that the figures obtained on clean laboratory surfaces can be maintained under industrial conditions. These are as follows:

(1) Vaporisation of the feed preferably not to exceed 80%.

(2) Maximum allowable heat flux for forced-circulation vaporisers and reboilers vaporising organics is 20,000 B.T.U./sq.ft.hr. and, for natural circulation vaporisers, 12,000 B.T.U./sq.ft.hr.

(3) Maximum allowable vaporising film coefficient for the forced or natural circulation vaporisation of organics is 300 B.T.U./sq.ft.hr.°F.

In the present case only the second condition is maintained and consequently in practice it is very probable that a coefficient of 300 B.T.U./sq.ft.hr.°F. is not exceeded and also that the vaporisation might not exceed 80%. However, the steam stripping ejector on the inlet to the separator provides complete flexibility and 100% stripping of the solvent with, however, a slightly increased consumption of stripping steam.

It is important to note that, in the present case, with heat fluxes approximating to only a tenth of the maximum allowable, the liquid heating coefficient is controlling and is therefore independent of the temperature difference up to a maximum figure of 60° to 120°F. (Drew and Mueller), figures which are not exceeded in practice in the present case.

A detailed sketch of the evaporator, steam stripping ejector and separator is given in Fig. 1, and a typical flowsheet for this stage of the process, which comprises the evaporation plant, settler and distillation columns, is given in Fig. 3.

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[13] Cessna, Lientz and Badger, *Ibid.*, 1940, **36**, 759.

Computer Control in Processing

is the subject of a special feature which will appear in the October issue of CHEMICAL AND PROCESS ENGINEERING. This feature will survey the usefulness and applicability of computers for process control and chemical plant design. The following articles are included in this feature:

Applications of Computers to Process Control
by J. F. Roth

The Use of Computers in Chemical Processes
by A. J. Longley

Design of Heat Exchangers by Digital Computer
by D. K. Houtby

Other articles appearing in next month's issue will be:

CPE Unit Operations Review—Filtration
by H. K. Suttle

Safety Assessments in the Nuclear Industry—Part 2
by A. Quinton

Materials of Construction for Chemical Plant—New Metals
by A. H. Barber

CPE DIARY

SEPTEMBER 5 TO 15 International **Packaging Exhibition** to be held at Olympia. Details from Press Officer, 34 Victoria Street, London, S.W.1.

SEPTEMBER 6 TO 8 Conference to discuss recent advances in **microwave measurement techniques** to be held at the Institution of Electrical Engineers, Savoy Place, London, W.C.2.

SEPTEMBER 18 TO 21 International Congress of **Food Science and Technology** to be held in London. Details from the Hon. Secretary, International Congress of Food Science and Technology, 14 Belgrave Square, London, S.W.1.

SEPTEMBER 21 TO 22 Conference on **Radio Spectroscopy of Solids** to be held at the University College of North Wales, Bangor. Details from the Institute of Physics, 47 Belgrave Square, London, S.W.1.

SEPTEMBER 25 TO 29 Eighth annual refresher course on **Current Practice in Fuel Efficiency** to be held at the Clarendon Laboratory, Oxford. Details from the Secretary, N.I.F.E.S., 181 King's Road, Reading.

SEPTEMBER 27 TO OCTOBER 4 International Conference on **Heating, Ventilation and Air Conditioning** to be held at Olympia. Details from Industrial Exhibitions Ltd., 9 Argyll Street, London, W.1.

SEPTEMBER 30 TO OCTOBER 7 Technical exhibition for **water supply, wastewater treatment and refuse disposal** to be held in the halls of the Swiss Industries Fair, Basle.

SEPTEMBER 30 TO OCTOBER 8 **MAC 61** (International Chemical Plant Exhibition) to be held at Milan. This is organised by the Italian Association of Chemists who will also have their annual meeting from OCTOBER 2 TO 5. Information from Secretary General, MAC 61, Via Desiderio da Settignano 13, Milan.

OCTOBER 3 TO 12 **Electronic Computer Exhibition** to be held at Olympia, London.

Personal Paragraphs

★ The permanent labour force of Wood, Pritchett & Partners Ltd. engaged on labour hire service, now exceeds 500 tradesmen. During the past four years the company has been actively associated with the construction and installation of micro-wave projects and nuclear missile bases. **Mr. W. F. Foulds** has negotiated contracts on behalf of the company.

★ Laporte Industries Ltd. announce that **Mr. B. H. Oldfield,** director and engineering manager of Laporte Chemicals Ltd., is to retire in September. He will be succeeded as engineering manager by **Mr. Maurice Ruddick.**

★ **Mr. L. Friend** has just been appointed director of chemical engineering development by the M. W. Kellogg Co., New York. In this capacity Mr. Friend will manage the pilot plant, process development and technical data groups of the company.

★ **Mr. G. P. Phillips** has recently been appointed to the main board of Prodorite Ltd. as sales director. He joined Prodorite in 1938 and was formerly general sales manager.

★ **Lt.-General Sir William Stratton,** C.B.E., D.S.O., has been appointed chairman of Edwin Danks & Co. (Oldbury) Ltd., and of Penman & Co. Ltd., in succession to the late **General Sir Kenneth Crawford.** Both these companies are members of the Babcock & Wilcox Group.

★ **Mr. J. H. Thornton** has been appointed group resident superintendent for the 580-MW nuclear power station at Sizewell, Suffolk, which is being built for the Central Electricity Generating Board by the English Electric, Babcock & Wilcox, Taylor Woodrow Atomic Power Group.

★ **Mr. P. E. Rousseau,** managing director of Sasol (South African Coal, Oil & Gas Corp. Ltd.), has now been appointed chairman of the corporation in succession to **Dr. F. J. Du Toit,** who died recently while on a trade mission to the Far East.

★ **Mr. F. Murphy** has recently joined Metal & Pipeline Endurance Ltd. as a senior cathodic protection engineer. His principal activities will be in connection with the various pipeline cathodic protection projects being carried out by Mapel.

Mr. L. Friend

Mr. W. F. Foulds

Mr. M. Ruddick

★ **Mr. R. M. Currie,** head of the Work Study Dept. of I.C.I., has been elected first president of the newly formed European Work Study Federation.

★ **Mr. L. H. Tearle,** chairman and managing director, Lawrence H. Tearle & Co. Ltd., has died at the age of 45. Mr. Tearle was at one time an executive at Leonard Hill Ltd., and personal assistant to Mr. Leonard Hill. About 12 years ago he started a group of technical journals in South Africa and later opened a London office from which he published *Grocery World* and *Handling International.*

We extend our sympathy to his widow and five children. His widow (formally Miss Joan Redhouse) was also for many years a valued employee of the Leonard Hill Group.

★ The council of the Institution of Chemical Engineers has elected the following to honorary membership in the class of corporate members: **Mr. H. W. Cremer,** C.B.E., **Prof. M. B. Donald,** F.R.I.C., **Mr. F. A. Greene** and **Sir Harold Hartley,** G.C.V.O., C.B.E., F.R.S.

★ **Miss Mary Goldring,** a former editor of the Leonard Hill Technical Group, was interviewed in a B.B.C. programme discussing the political and technical implications of the space flight of Major Titov in the Russian Vostok II spaceship.

★ **Mr. S. R. Badley** has been appointed a director of Extrudex Ltd.

New Books

Radioactive Contamination of Materials Used in Scientific Research. By J. R. DeVoe. Publication 895. National Academy of Sciences—National Research Council, Washington, D.C., 1961. Pp. vii + 142.

The potential danger to health of ionising radiations from radioactive substances has frequently been emphasised, but it should also be generally appreciated that radioactive contamination may have a deleterious effect on scientific studies involving low-level counting. To obtain information concerning this latter problem, the sub-committee on radiochemistry of the National Academy of Sciences-National Research Council of the U.S.A. engaged Dr. DeVoe as a consultant. The data obtained as a result of his nation-wide investigation are collected and discussed in this nuclear science series report.

While the number of scientists whose work is liable to be adversely affected by radioactive contamination is relatively small at present, there is every indication that the demand for uncontaminated materials and chemical reagents will increase in the future. Also, since the information on the amount of radioactive contamination is limited and scattered, the present report is to be welcomed as providing an authoritative survey of the problem. An indication of the thoroughness of Dr. DeVoe's investigation is provided by the fact that 130 scientists throughout North America were consulted. The names and addresses of these experts are listed in an appendix.

The data collected in the report show that one substance, krypton, has been contaminated to an extent which has seriously curtailed some of its possible scientific uses. The contaminant in this case is fission product Kr^{85} (half-life 10.6 years) which has escaped from its environment and contaminated the krypton of the atmosphere. This probably could not have been prevented but, if the seriousness of the situation had been foreseen, adequate steps could have been taken to stockpile sufficient uncontaminated gas. This most graphic example of radioactive contamination illustrates the need to stockpile materials which are now uncontaminated. The cost of such a programme may be large, but not greater than the cost of purification of a substance if it has become radioisotopically contaminated.

Another example of contamination mentioned in the report, and with which the reviewer has been concerned, is the contamination of ruthenium produced in recent years with fission product Ru^{106} (half-life one year). In this case, the manufacturer has been willing to co-operate with the research scientist, and all stocks of ruthenium produced by the International Nickel Co. (Mond) Ltd. have been monitored and only inactive material is marketed. The problem of radioactively contaminated ruthenium is not very serious, but is important enough to be called to the attention of scientists.

The report emphasises that process changes could avoid many radioisotopic contaminations. In some cases, *e.g.* that of krypton, the contamination may not be easily avoided; therefore it may be advisable to accelerate research on isotopic separations. In some cases, commercial manufacturers as well as scientific research workers are concerned about low levels of radioactive impurities in the materials they use. This is particularly the case in the photographic industry. The report emphasises that manufacturers of crystals for radiation detectors must exclude radioactivity from their products. Manufacturers of photomultiplier tubes must also take care to exclude radioactivity from all internal parts of the tubes as well as from the glass envelopes.

Publication 895 finishes with an addendum giving recommendations by the sub-committee on radiochemistry of the National Academy of Sciences-National Research Council. Altogether the project has been very worth while. D. F. C. MORRIS

Telomerization and New Synthetic Materials. By R. Kh. Freidlina and Sh. A. Karapetyan. Pergamon Press, 1961. Pp. 102. 25s. net.

Telomerization is a relatively new method of synthesising organic compounds developed in 1942 by the American chemists Hanford and Joyce to describe the following reaction:

$$XY + nCR_1R_2 = CR_3R_4 \rightarrow X(CR_1R_2CR_3R_4)_nY$$

where the polymerisable monomer is called ' taxogen ' and the molecule XY from which the end groups are derived is called ' telogen '. The simplest case illustrating this reaction is where R_1, R_2, R_3 and R_4 are all H atoms and XY represents CCl_4. This, in fact, is a mixture of telomers derived from ethylene which are synthesised by free radical mechanisms involving $Cl^{\cdot}$ and $CCl_3^{\cdot}$.

Tetrachloralkanes having a structure $Cl(CH_2—CH_2)_nCCl_3$ were actually produced by telomerization in a pilot plant at the Russian Institute of Elemento-Organic Compounds. The process involved reacting an excess of ethylene with carbon tetrachloride at a pressure of about 50 atm. By varying the pressure the number of C atoms in the tetrachloralkanes could be changed. It is relatively simple to obtain various organic intermediates from these tetrachloralkanes and the authors have described in detail how they obtained amino heptylic acid which can be polymerised to give a polyamide fibre ' enanth ', related structurally to nylon-6 from caprolactam but having superior physical properties to nylon-6. This example is given to illustrate the usefulness of telomerization where organic compounds having, for example, 7 C atoms can be reasily synthesised.

Obviously from tetrachloralkanes other compounds can be produced as for example chloracids, hydroxyacids, ω-amino acids, dicarboxylic acids and cyclic lactones. Each of these compounds plays a very important role in organic chemistry and their large-scale production becomes increasingly important in the contemporary chemical industry.

This book is to be recommended, if only for the insight it can give to this fascinating new branch of organic chemistry. Unfortunately, the authors have assumed that the reader has little chemical background and have therefore simplified and explained every concept in organic chemistry quite needlessly. It would have been far more beneficial to prune down the contents and omit chapters dealing with polymerisation and fibre tech-technology—both of which are far better explained in dozens of existing English textbooks. The poor production by photo-lithography and non-letterpress setting makes the cost of this slim volume (25s.) rather excessive. The translation is generally of a very good standard; quite often the translator must have been hampered by having to adhere closely to a Russian text that was badly edited.

I.L.H.

What's New

in Plant • Equipment • Materials • Processes

CPE reference numbers are appended to all items appearing in these pages to make it easy for readers to obtain quickly, and free of charge, full details of any equipment, machinery, materials, processes, etc., in which they are interested. Simply fill in the top postcard attached, giving the appropriate reference number(s), and post it.

Insulating material

Johns-Manville *Thermobestos*, an inorganic insulating material, is composed of asbestos, diatomaceous silica and lime bonded with strong asbestos fibres. After the initial moulding, the insulation is subjected to a special steam process. Live steam converts the lime and diatomaceous silica into hydrous calcium silicate, a substance noted for its tough composition. The resultant inorganic insulating material has low thermal conductivity, great structural strength and insolubility in water, is light in weight and suitable for high-temperature process equipment which must withstand severe outdoor operating conditions.

CPE 1716

Heating tapes

Isopad Ltd. will be displaying electrical heating tapes at the Heating, Ventilating and Air Conditioning Exhibition. This will include flexible heating tapes used for the tracing of fuel oil lines from 200 sec. viscosity upwards. The heating tapes are made in standard lengths from 18 in. to 270 ft. and come in a variety of loadings from 5 W/ft. for frost protection, to 70 W/ft. for high-temperature applications. A full range of thermostatic controls are available.

Amongst their latest developments is a metal-sheathed heating element mounted on stainless-steel mesh, thus offering a robust appliance suitable for temperatures up to 600°C.

CPE 1717

Steam equipment

Midland Industries Ltd. will exhibit at the Heating, Ventilating and Air Conditioning Exhibition their full range of steam equipment, which includes their thermostatic and float-operated range of steam traps. The latest addition to this range is the *Mil* thermatic (M.T.M.) trap.

Reducing valves and packless gland valves, together with the remainder of the *Mil* range of products, accounted for their other exhibits. The packless gland valve has a stainless-steel bellows in place of the normal gland, and the rate of flow through the valve can be regulated by an adjusting screw which is independent of the hand wheel.

CPE 1718

Temperature controller

A new mercury-in-steel, on-off temperature controller, which has independent circuits allowing control at any two points through one instrument, has been produced by the British Rototherm Co. Ltd.

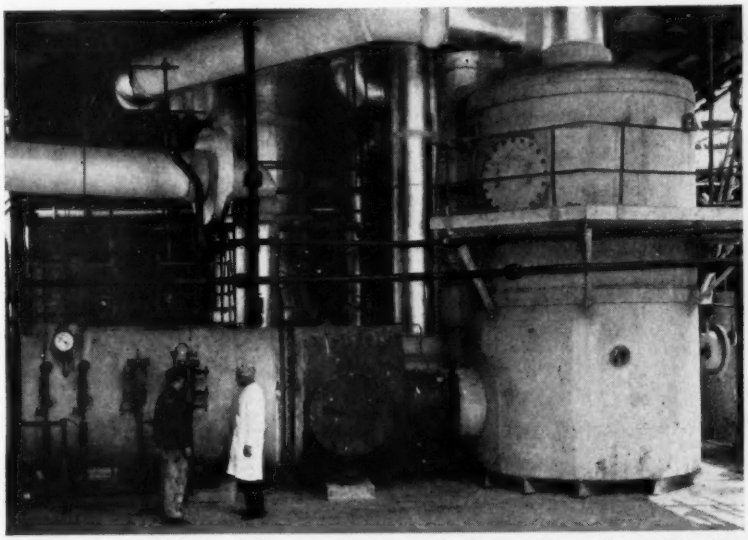
Each of the two controller circuits consists of alloy steel Bourdon tubes, capillaries and temperature-sensitive bulbs, and operating mercury switches rated up to 30 A at 230/250 a.c. The differential is fixed but varies according to switching capacity. Indicator lamps and bottom-entry electrical conduit are standard.

Temperature ranges are available between −20° and +1,000°F. (minimum coverage 100°F.) and the equivalent °C. **CPE 1719**

Flowrators

Fischer & Porter Ltd., makers of area flowrators, have improved their range of ' snap-in ' glass tube flowrators.

The company are now making their glass metering tubes with flame-polished ends, which reduces the possibility of accidentally chipping the tube and makes the ends round and smooth in section, giving them additional strength. **CPE 1720**



A contact process sulphuric-acid plant designed by F. W. Berk and Simon Carves Ltd. which incorporates a modern fire tube boiler and a four-pass converter **CPE 1721**

Sewage purification

A new process has been developed by Th. Hoelscher Maschinenfabrik G. Neidl which improves the performance of sludge and sewage purification plants.

By means of a weak electric current which is passed into the sewage, the micro-organisms are induced to accelerate their metabolism. The two electrodes are fed from an a.c. source with weak electric current at regular intervals of time. Although the distance between the electrodes is only 2 ft., the electric stimulation is transmitted throughout the effluent tank.

The technique, which is both simple and economical, has already been applied in several cases. In Strasbourg, for example, a sewage plant which was already in a state of advanced putrefaction was clarified and made sanitary. By means of this technique it is also possible to accelerate other fermentation processes such as maturing of whisky, ripening of cheeses, etc. **CPE 1722**

Gas-analysis instruments

Elliott Bros. (London) Ltd., a member of the Elliott-Automation Group, have negotiated with the Office National d'Études et de Recherches Aeronautiques (O.N.E.R.A.) of France a licence to manufacture and sell two gas - analysis instruments of very advanced design. The analysers are of the paramagnetic type, a principle used in both thermo-pneumatic and thermo-magnetic convection systems of measurement.

The new instruments are primarily intended for the determination of oxygen content in gaseous mixtures and have already found application in the control of furnace atmospheres in processes such as cement manufacture, porcelain kilns and steel founding. Catalyst protection, flue-gas analysis and the control of atmospheric test rooms are further examples of their use. The instruments have an important part to play in the fast-growing field of continuous quality control and in the attainment of higher operating efficiencies in process plant of all types. **CPE 1723**

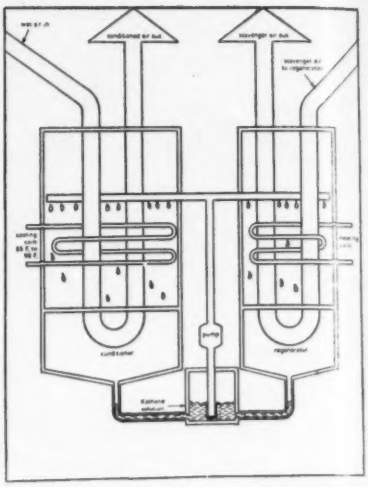
Dust-measuring equipment

On the A.E.I. stand at the Clean Air Exhibition, the company's Electronic Apparatus Division will demonstrate the A.E.I. dust-measuring equipment for power stations and processes. This has been designed to overcome certain disadvantages in other methods of recording the density of smoke discharged through a duct or chimney.

To prevent condensation of moisture in the sampling equipment, the temperature of working parts is kept above dewpoint. Build-up of dust is prevented by automatically purging with air for a few seconds once every 20 min. The equipment, being highly sensitive, will respond to particle diameters ranging down to 0.01 micron, and can monitor flue gases leaving dust-arresting plant of large boilers with burdens of about 0.04 gr./cu.ft., rising to 2 gr./cu.ft. during soot blowing. **CPE 1724**

Humidity conditioning system

The Chemical Engineering Division of W. C. Holmes & Co. Ltd. has obtained a licence from the International Engineering & Trading Society N.V., of Holland, to market the *Kathabar* humidity conditioning system in the U.K. This system, which was developed by the Surface Combustion Corp., U.S.A., is based on the drying properties of kathene, a liquid absorbent of which lithium chloride is the main constituent.



The 'Kathabar' humidity conditioning system

A *Kathabar* unit consists essentially of a drying tower containing cooling coils, a regenerator tower containing heating coils, and a pumping unit and drainage tank. By regulating the temperature of the solution, it is possible to control the humidity of the air.

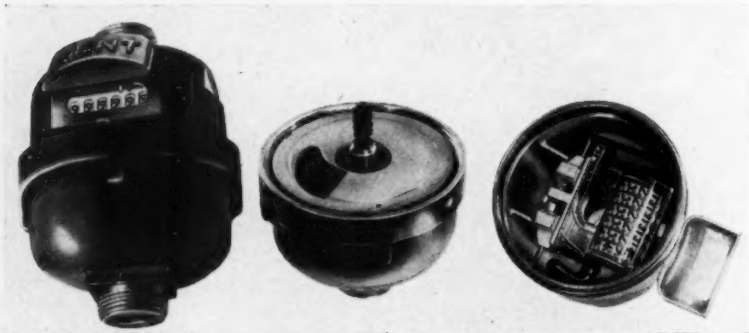
Dehumidification is a continuous process with no reversing. The operation of the unit is fully automatic and the kathene solution need not be replaced before at least five years of continuous operation.

Kathabar units are suitable for many applications in a wide variety of industries, particularly where sterile air is desirable. **CPE 1726**

Synthetic mono-carboxylic acids

Developed by the Shell laboratories in Amsterdam, a new group of synthetic organic acids is now available from a pilot plant and will be produced on a commercial scale as from January next. The acids are being marketed under the trade name *Versatic*. Although similar in some respects to other organic acids, the new acids possess properties which will find important applications in the surface coatings, plastics, rubber, metallurgical and many other industries.

Versatic acids are produced from olefins, carbon monoxide and water. It is expected that eventually a full range will be made available. At present, however, the acids are a blend of C_9, C_{10} and C_{11} mono carboxylic acids. Research into applications and products derived from *Versatic* acids



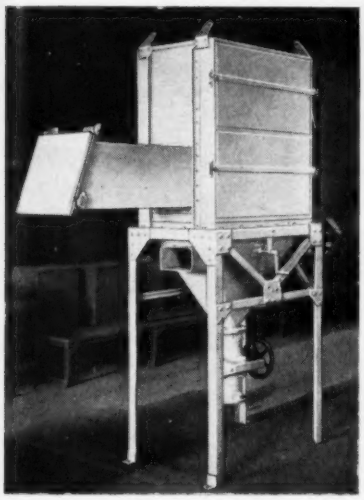
On the left is the new Kent ½-in. SM (ebonite) water meter with flap raised to show the straight reading counter. In the centre and to the right are halves of a Kent ½-in. SM water meter unscrewed to show the working chamber and worm drive and (right) the combined reduction gear and counter unit, located by a sprung wedge **CPE 1725**

is currently being carried out. Fields of interest include emulsifiers for rubber and plastics, plasticisers, foam promoters, wood preservatives, and the amines and amides. **CPE 1727**

Dust collectors

Dallow Lambert Ltd. have incorporated certain features in their *Unimaster* dust collectors to offset the tendency of the material to arch when in a repose position in the hopper, and to eliminate possible explosion hazards.

The plant has its inspection doors strengthened and an extended explosion relief duct is fitted to be taken



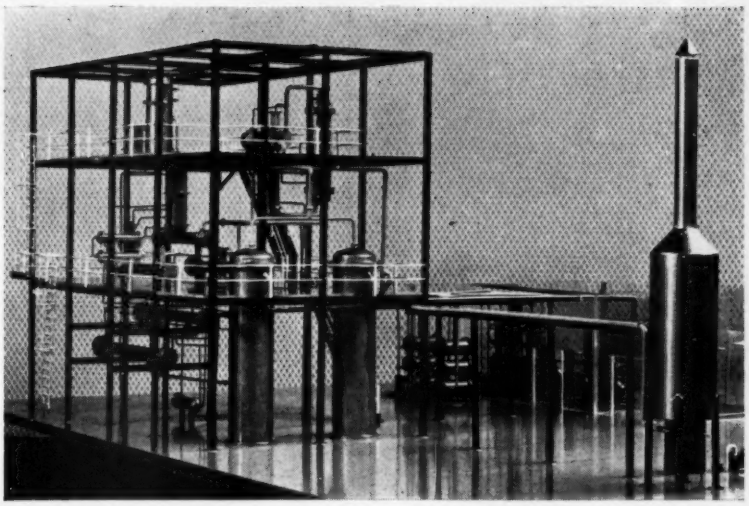
The Dallow Lambert 'Unimaster' dust collector

through an adjacent wall. The hinged relief door has a limit switch which automatically trips the fan motor if it is open, thereby safeguarding against further entrainment of dust and a possible secondary explosion. A hand-operated cranked lever is fitted in the top of the hopper, and the collected dust is discharged downwards back into the system by means of a 6-in.-diam. rubber tube pinch valve. **CPE 1728**

Polyurethane rubber

I.C.I. have introduced a new *Daltoflex* polyurethane rubber designed for processing on conventional rubber machinery. It is cured by means of *Suprasec* M, a liquid isocyanate compound of high boiling point whose low vapour pressure ensures freedom from any toxic hazard during processing. Hard rubbers having good properties are obtained.

Unlike natural or other synthetic rubbers, an accelerator is not essential for curing; the cured compounds obtained are superior to natural and earlier synthetic rubber in tensile and tear strength, abrasion resistance, solvent resistance and in resistance to oxidation. **CPE 1729**



A model of a Newton Chambers design for a hydro-refining plant to be shown at the French Trade Fair in Moscow next August. Newton Chambers have world rights for the manufacture of plant to operate a process of hydro-refining crude benzole, naphthas and other hydrocarbon oils **CPE 1731**

Magnesium oxide

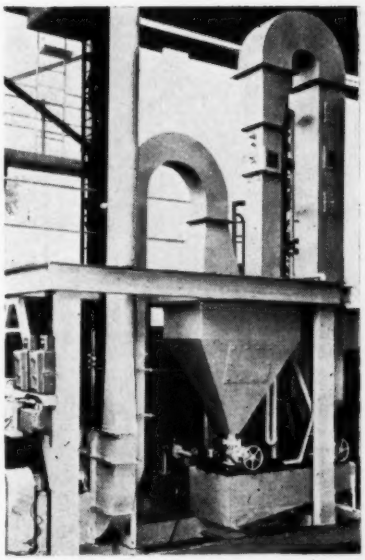
A new ceramic material described as combining great purity and density has been developed by research scientists at the American associates of Honeywell Controls Ltd. The material is a translucent magnesium oxide which, when chemically polished, is said to have a strength of 45,000 p.s.i., whilst that of ordinary magnesium oxide has a strength of 24,000 p.s.i. In addition to the greater strength possessed by the new material, it has a density which comes within 2% of the possible maximum, giving better finishes.

The new ceramic material has a melting point of 2,800°C. and could be used as the heat-exchange medium in applications such as wind-tunnel pebble-bed heaters, or electrical insulation at high temperature. **CPE 1730**

Rubber-debonding plant

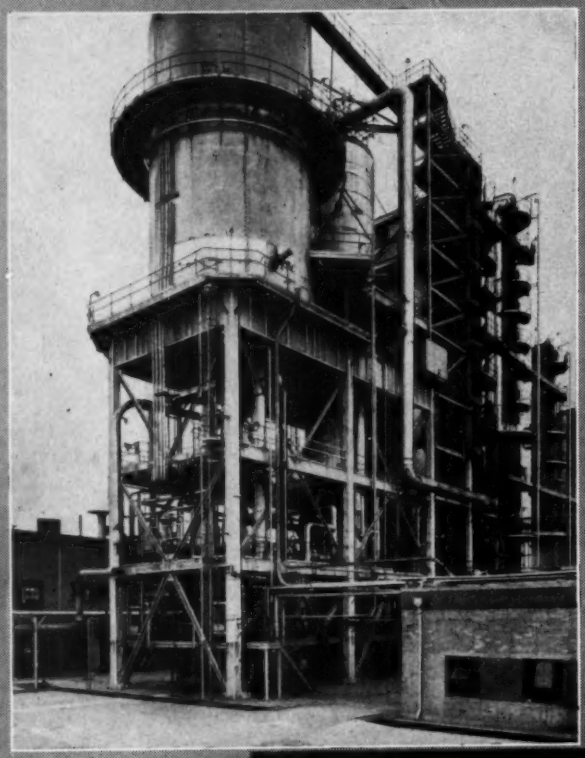
The latest Holmes-Andre debonding plant, which operates on the principle of destructive distillation in a closed vessel, incorporates new design features.

A double-ended 1-h.p. motor which drives the air blower also drives a pump which circulates distillate oil throughout the system. This not only irrigates the offtake piping but also washes the rubber vapour in a scrubber cooler and so maintains a constant level in a seal tank. A built-in rodding device is incorporated for the short section of pipe that cannot be irrigated. The addition of a small-bore low-pressure steam supply enables the retort to be emptied whilst hot, thus reducing operating times. **CPE 1732**



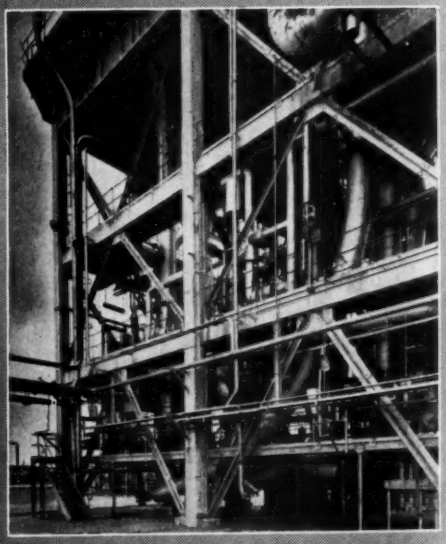
The recently commissioned portable pilot plant which will be used by Ambuco Ltd. to establish optimum working conditions for their 'Venturi' scrubbers which are being installed for dust removal from industrial fumes **CPE 1733**

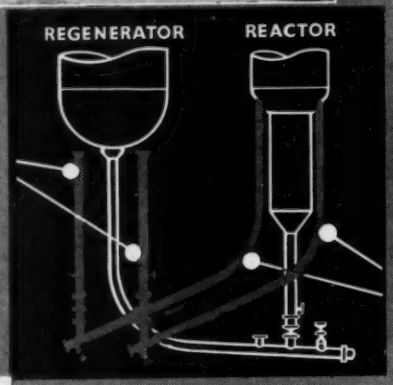
PIPEWORK
FOR PETROLEUM PLANT



At the Grangemouth Refinery of B.P. Ltd., John Thompson (Pipework) Ltd., supplied the catalyst feed pipe lines from the regenerator, and the crude oil and catalyst mixture feed pipe lines to the reactor.

These lines which serve an integral part of the process have been supplied to B.P. Ltd., over a period of up to 10 years, and are completely fabricated to Class 1 specification of high grade mild steel for a temperature of 970°F at 30 lb/sq. at the Wolverhampton works of John Thompson (Pipework) Ltd.





Catalyst feed lines 18 in. diameter.

Crude residual oil and catalyst mixture feed lines to reactor. 20 in. diameter.

JOHN THOMPSON (PIPEWORK) LIMITED
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JTP/11

Nuclear Notes

C.E.R.N. annual report

Progress made in 1960 by the first joint European establishment for scientific co-operation—the nuclear research laboratory at Meyrin, near Geneva—is dealt with in the annual report recently issued by C.E.R.N.

Graphite examination

At Calder Hall, U.K.A.E.A. engineers have been examining what effects 4½ years' irradiation has had on their graphite moderator. A Pye miniature TV camera, only 3 in. diam., was developed to operate inside the core, and an equally small tool was developed to cut pieces of graphite out of the reactor for inspection. The cutting tool was attached to the end of 50 ft. of hose carrying the circuits, by which the tool was operated remotely. The cut pieces gave the same satisfactory results as the Wigner gap measurements.

Greek research reactor

Designed and built by American Machine & Foundry Co., the 1,000-kW nuclear research reactor is located on the outskirts of Athens. The new Democritus Nuclear Research Centre will serve as a research facility for scientists and students and will be run by the Atomic Energy Commission of Greece. The U.S. Atomic Energy Commission will give the Greek A.E.C. $350,000 toward the cost of the reactor under the U.S. Government's 'Atoms for Peace' programme.

New research centre

An agreement between Euratom and the Dutch Government was recently signed in Brussels. This will enable the Community to establish part of its Common Nuclear Research Centre at Petten, near Amsterdam.

Under the agreement the Dutch Government transfers to Euratom the high-flux reactor at Petten and land which adjoins it, which have hitherto formed part of the Netherlands Nuclear Research Centre.

The Petten Research Centre, which is the fourth to be established by the Community, will be concerned with general nuclear research, but in the early stages, work will be concentrated on the high-flux reactor, which is due to go critical towards the end of the current year.

Plastic reactor facility

Initial criticality has just been achieved in a plastic-moderated low-power reactor designed and built at Battelle Memorial Institute in the study of a new energy-conversion process. The unit is being operated by the Columbus, Ohio, research centre under contract with Pratt & Whitney Aircraft Co.

Radiation generated by the reactor, a tube-like construction, is being used in the study of a process in which the kinetic energy of the fragments resulting from fission of uranium is converted directly into electricity. The experiments requiring high radiation levels are continuing at the research reactor, while experiments requiring combinations of radiation, high vacuum and special equipment arrangement, are scheduled for the plastic reactor facility.

Nuclear power plant

A 325,000-kW nuclear power plant to be built at Bodega Bay, near San Francisco, by Pacific Gas & Electric Co. will incorporate new developments in boiling-water reactor technology made by U.S. General Electric.

It is said that the nuclear system for the plant will have the largest single-cycle boiling-water reactor ever built and will be simpler and more economical than earlier reactors. General Electric will design and supply the reactor, its nuclear steam supply equipment and the enriched uranium nuclear fuel elements for the plant. Average life of the first core will be about 3½ years.

Construction of the $59-million plant is expected to start in August 1962, with completion scheduled for late 1965.

DRAGON annual report

The O.E.E.C. European Nuclear Energy Agency has published the second annual report to its Steering Committee on the DRAGON high-temperature reactor project at Winfrith. It deals with the progress during the year under review, and is in three sections. The first two (research and development, and reactor design and construction) cover in some detail the progress of work in the two Technical Divisions of the project. The remaining section (administration) deals with staff and contractual matters, finance, information and patents, and co-operation with the U.S. Atomic Energy Commission in respect of the Peach Bottom H.T.G.C. reactor project.

Moscow biochemistry congress

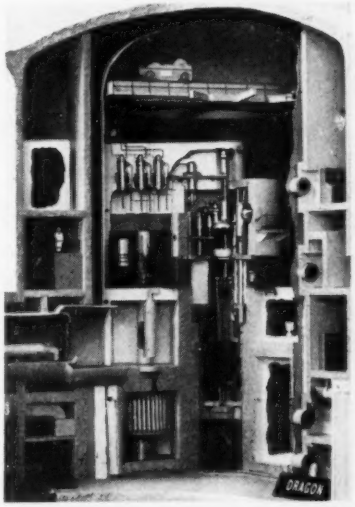
The International Atomic Energy Agency has taken an active part in the fifth International Congress of Biochemistry, held in Moscow in August.

It shared the cost of the congress section dealing with radio-biochemistry and was represented by Dr. H. Seligman, deputy director-general in charge of the Department of Research and Isotopes, Dr. Bernhard Gross, director of the Division of Scientific and Technical Information, and Dr. H. Vetter and Dr. V. Zeleny, both from the Agency's Division of Isotopes.

Director General of I.A.E.A.

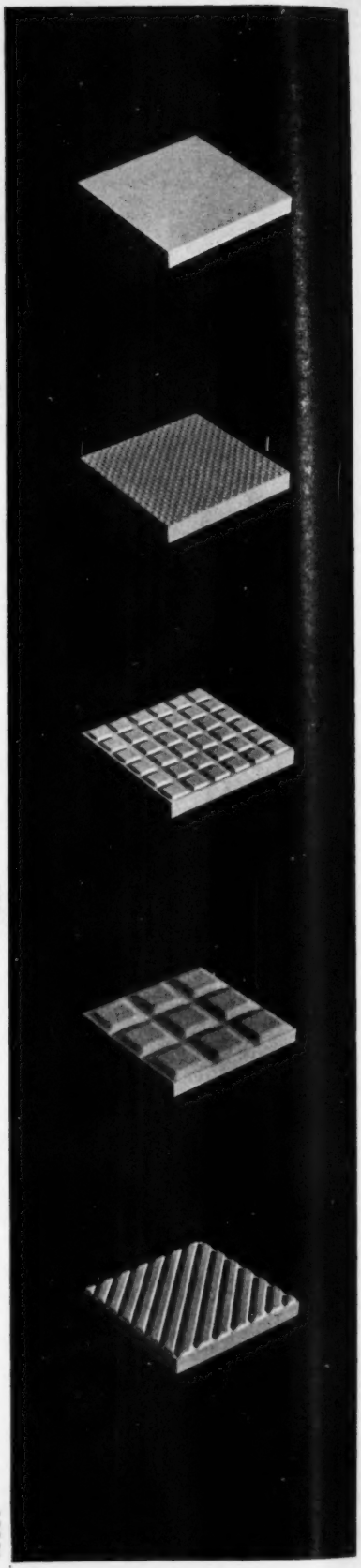
Dr. A. S. Eklund has been appointed by the board of governors of the I.A.E.A. to the post of director general, subject to approval by the I.A.E.A.'s general conference.

Dr. Eklund acted as secretary-general to the second U.N. Geneva Conference on the Peaceful Uses of Atomic Energy in 1958, and is chairman of the international board of management of the O.E.E.C. high-temperature reactor project DRAGON at Winfrith.



A cutaway model of the DRAGON reactor being built by U.K.A.E.A. Winfrith

LEFCO



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40 Buckingham Palace Road London SW 1

PRODORITE LIMITED
Eagle Works Wednesbury Staffordshire

SEMTEX LIMITED
50 Blackfriars Road Salford 3 Lancashire
also
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CPE Company News

Oxygen and nitrogen plants

Constructors John Brown Ltd. were recently awarded a contract worth nearly £2 million by Polimex, the Polish Government trade organisation.

This contract is for the design, supply and installation of three tonnage oxygen and nitrogen plants which are to be used in the production of ammonia at Tarnow in the south of Poland. The plants will be completed successively within a period of about five years and they will have a combined output of nearly 1,000 tons/day of oxygen and more than 1,100 tons/day of nitrogen.

Acid shipment

A cargo of nearly 1,000 tons of sulphuric acid was recently shipped to the Continent, the first under a multi-thousand-ton contract gained by Solway Chemicals Ltd., a member of the Albright & Wilson Group. The acid is not made from imported sulphur but from an indigenous raw material, anhydrite, mined at Whitehaven.

New welding research laboratory

Lord Mills, a former Minister of Fuel and Power, recently opened the new 21,000-sq.ft. British Welding Research Association's engineering laboratory at Abington. The ceremony began a two-day exhibition of research in progress. In addition to developments in conventional welding techniques and investigations shown, a friction welder built by the Association was demonstrated.

De-alkalisation base exchange plant

William Boby & Co. have been awarded contracts by British Glues & Chemicals Ltd. for the supply of two de-alkalisation base exchange plants. The total value of the orders is more than £10,000.

The plants will be installed at the Luton and Bermondsey works, and will have capacities of 3,000 and 4,000 gal./hr. respectively.

Synthetic fibre processes

An agreement has been reached by Humphreys & Glasgow Ltd. with East Germany covering know-how and licences for a wide range of processes in the East German chemical industry. These include their up-to-date processes for the production and spinning of polyacrylonitrile, polyamide, polyester and viscose fibres.

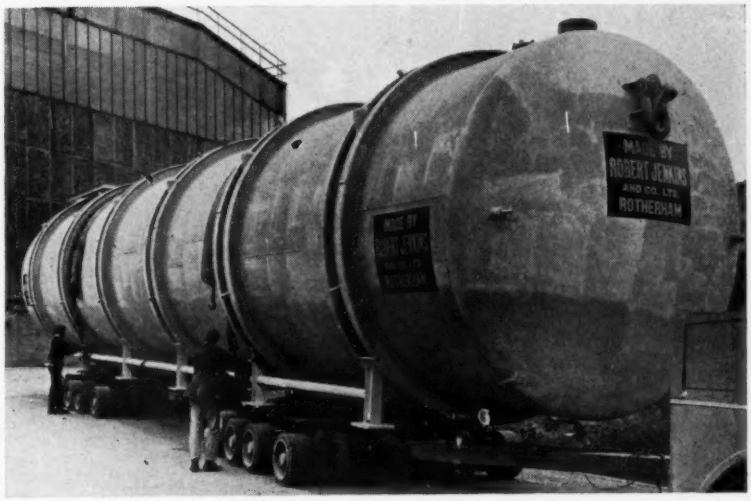
Liaison office

W. R. Grace & Co. are to establish a Paris office which is expected to facilitate technological contacts between European companies and Grace Chemical Divisions at home. The new office will not interfere with Grace chemical manufacturing plants in Europe, which operate under the control of the Overseas Chemical Division. This division operates six plants in Europe as well as others in various parts of the world.

Expansion in America

Courtaulds Ltd. have completed the first step in plans for the exploration of areas of mutual interest with Koppers Co. Inc.

In October 1959, Courtaulds announced the formation of a new subsidiary company, Courtaulds North America Inc., to explore and develop opportunities for the expansion of their activities in American industries, outside textiles. Courtaulds have recently acquired an interest amounting to some 4% of the common stock of Koppers, discussions taking place regarding the fields outside textiles in which technical, process and production knowledge might be exchanged.



Three-million-cu.ft. capacity liquid oxygen vacuum insulated storage vessel, manufactured for, and jointly designed with the British Oxygen Co. Ltd., ready for its journey from the works of Robert Jenkins & Co. Ltd. for installation at the South Durham Co.'s steelworks at West Hartlepool

Measuring equipment

George Kent Ltd. have received further orders for instrumentation from the Polish national importing organisation, Varimex. The special equipment ordered is valued at £30,000 and is used for the measurement and control of processes in the Polish chemical industry.

£ s d

CHEMICAL PLANT COSTS

Cost indices for the month of July 1961 are as follows:

Plant Construction Index: 185.5

Equipment Cost Index: 174.3

(June 1949 = 100)

£ s d

Coal preparation plant

Simon-Carves Ltd. have received a contract to design and erect a dense medium coal-preparation plant at Bentley Colliery in the North Eastern Division of the National Coal Board.

The plant will provide additional large coal-cleaning facilities and will be capable of washing 250 tons/hr.

Oilseed extraction plant

Blaw Knox Chemical Engineering Co. Ltd. have been awarded a contract by J. Bibby & Sons Ltd. to furnish a plant for the solvent extraction of soya beans and other oilseeds. The plant, to be constructed at Liverpool, will supersede an existing solvent extraction plant, and the programme is scheduled for completion by March 1962.

CPE World News

JAPAN

Reforming unit

A *Houdriformer* unit with a nominal design capacity of 6,000 bbl./stream day of petroleum naphtha has started operations at the Nippon Mining Co.'s refinery at Mizushima, Japan.

This *Houdriformer*, licensed and designed by Houdry Process Corp. of Philadelphia, is the largest of its kind in Japan. The unit has a pre-treater which also handles the desulphurisation of naphtha to be used in jet plane fuel. This reforming installation uses high-paraffin, high-sulphur naphtha derived from Middle East crude oil and transforms it into a completely desulphurised 95 clear octane gasoline.

KUWAIT

Chemicals production

An agreement for the production of chemicals in Kuwait was recently signed by the Kuwait Government and the De Nora Co. of Milan. As a result a new company will produce caustic soda, raw materials for plastics, oxygen, acetylene, chlorine and nitrogen fertilisers. The enterprise will utilise natural gas for power and for the production of chemicals.

UNITED STATES

Phthalic anhydride plant

Allied Chemical Corp. will begin construction of a plant to manufacture phthalic anhydride on the site of its present plant at El Segundo, near Los Angeles.

The plant will employ the Von Heyden-Chemiebau process, which was selected because it can utilise a variety of raw materials. The new facilities, which are scheduled to be in production by the latter half of 1962, will supply the growing requirements for phthalic anhydride on the West Coast of the U.S. The construction contract has been awarded to Ralph M. Parsons Co. of Los Angeles, California.

CANADA

Petrochemical expansion

Preliminary work will commence shortly on construction of a $20-million petrochemical plant by Shawinigan Chemicals Ltd. and an associated $45-million 300,000-kW. thermal electric station by the Shawinigan Water & Power Co. on the south shore of the St. Lawrence River near Montreal. The petrochemical plant will be located at Varennes and is expected to start operation in mid-1963.

The British American Oil Co. Ltd. will process crude oil to provide the raw materials for the petrochemical plant and at the same time heavy fuel oil for the thermal station. Products of the petrochemical plant will be mainly ethylene and propylene.

SICILY

Antibiotics expansion

Plans to build a second major antibiotics plant in Europe have been announced by Cyanamid International. The new plant will be built at the Catania, Sicily, headquarters of Cyanamid Italia. The new production unit will represent an additional capital investment of approximately $1 million.

This new facility by Cyanamid Italia will be the company's first antibiotic fermentation unit within the European Common Market. Like its British equivalent at Gosport, the Catania plant will produce the main broad spectrum of antibiotics, ledermycin (demethyl chlortetracycline), achromycin (tetracycline) and aureomycin (chlortetracycline) as well as various other therapeutic agents.

YUGOSLAVIA

Synthesis gas production

Société Belge de l'Azote et des Produits Chimiques du Marly (S.B.A.), Liege, Belgium, and D. Bonaldi & Co. SpA., Italy, have concluded an agreement to supply the Yugoslav firm Proizvodnja Nafte with a plant for the production of methanol synthesis gas from natural gas.

This plant is designed by the Engineering Division of S.B.A. and operates a process called 'S.B.A.-H.T. process', owned jointly by S.B.A. and the Topsoe Co., Denmark. This process produces synthesis gas for ammonia and methanol by cracking various hydrocarbon stocks.

FRANCE

Ammonium nitrate plant

The French company Ammonia S.A. has just commissioned the Société Belge de l'Azote et des Produits Chimiques du Marly of Liege, Belgium, with the engineering of a plant for the production of granulated ammonium nitrate.

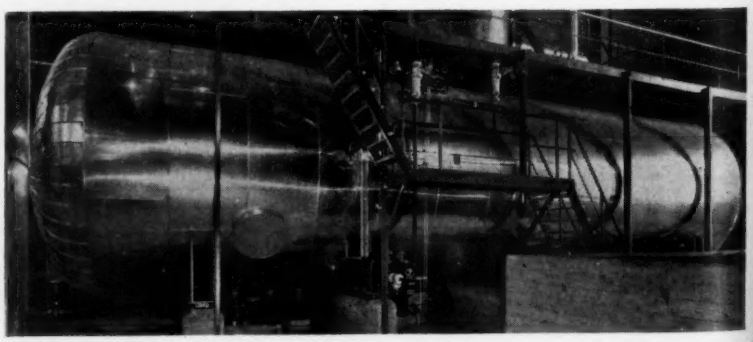
This plant will form part of the Wingles plant, Pas-de-Calais, of Ammonia S.A. It will have a daily capacity of 150 tons ammonium nitrate.

SPAIN

Petrochemical complex

At the annual general meeting of Dow-Uniquinesa S.A., held recently in Spain, it was announced that the production capacity of the permit granted by the Spanish Government to the company for a projected petrochemical complex will be 10,000 tons of high-pressure polyethylene, 6,000 tons of low-pressure polyethylene, 12,000 tons of styrene monomer and 8,000 tons of polypropylene. The new plant will be located in Somorrostro, near Bilbao.

Dow-Unquinesa is an associated company of the Dow Chemical Co.



The deaerator, designed by William Boby & Co., at the new Finnish paper mill at Heinola. The horizontal storage tank holds a 30-min. supply of water—175,000 lb. Above it is the deaerator dome